Pacific Northwest National Laboratory

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TANK VAPOR CHARACTERIZATION PROJECT

Headspace Vapor Characterization of Hanford Waste Tank 241-S-102: Second Comparison Study Results from Samples Collected on 04/04/96

K. H. Pool
J. C. Evans
B. L. Thomas

K. B. Olsen
J. S. Fruchter
K. L. Silvers

January 1997

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Prepared for Westinghouse Hanford Company
under a Related Services Agreement
with the U.S. Department of Energy
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Summary

This report describes the analytical results of vapor samples taken from the headspace of waste storage tank 241-S-102 (Tank S-102) at the Hanford Site in Washington State. The results described in this report is the second in a series comparing vapor sampling of the tank headspace using the Vapor Sampling System (VSS) and In Situ Vapor Sampling (ISVS) system without high efficiency particulate air (HEPA) prefiltration. The results include air concentrations of water (H_2O) and ammonia (NH_3), permanent gases, total non-methane organic compounds (TO-12), and individual organic analytes collected in SUMMA™ canisters and on triple sorbent traps (TSTs). Samples were collected by Westinghouse Hanford Company (WHC) and analyzed by Pacific Northwest National Laboratory (PNNL). Analyses were performed by the Vapor Analytical Laboratory (VAL) at PNNL. Analyte concentrations were based on analytical results and, where appropriate, sample volume measurements provided by WHC.

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Glossary

CCV	continuing calibration verification
COC	chain-of-custody
DIW	deionized water
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
GC/FID	gas chromatography/flame ionization detector
GC/MS	gas chromatograph/mass spectrometer
GC/TCD	gas chromatography/thermal conductivity detection
HEPA	high efficiency particulate air
IDL	instrument detection limit
IS	internal standard
ISVS	In Situ Vapor Sampling
LLS	low level standard
MDL	method detection limit
NIST	National Institute for Standards and Technology
% D	percent difference
PNL	previous designation for Pacific Northwest Laboratory
PNNL	Pacific Northwest National Laboratory
ppbv	part per billion by volume
ppm	parts per million
ppmv	part per million by volume
QA	quality assurance
QC	quality control
RPD	relative percent difference
RSD	relative standard deviation
SAP	sample and analysis plan
ST DEV	standard deviation
STP	standard temperature and pressure
SUMMA™	stainless steel, passivated interior canister
TBP	tributyl phosphate
TIC	tentatively identified compound
TNMOC	total non-methane organic compounds
TST	triple sorbent trap
UHP	ultra high purity
VAL	Vapor Analytical Laboratory
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

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1.0 Introduction

This report describes the results of vapor samples obtained to compare vapor sampling of the tank headspace using the Vapor Sampling System (VSS) and In Situ Vapor Sampling System (ISVS) without particulate prefiltration. Samples were collected from the headspace of waste storage tank 241-S-102 (Tank S-102) at the Hanford Site in Washington State. Pacific Northwest National Laboratory (PNNL)^(a) was contracted by Westinghouse Hanford Company (WHC) to provide sampling devices and analyze samples for water, ammonia, permanent gases, total nonmethane hydrocarbons (TNMOCs, also known as TO-12), and organic analytes in samples collected in SUMMA™ canisters and on triple sorbent traps (TSTs) from the tank headspace. The analytical work was performed by the PNNL Vapor Analytical Laboratory (VAL) by the Tank Vapor Characterization Project. Work performed was based on a sampling and analysis plan (SAP) prepared by WHC. The SAP provided job-specific instructions for samples, analyses, and reporting. The SAP for this sample job was "Sampling and Analysis Plan for Tank Vapor Sampling Comparison Test" (Homi 1996), and the sample jobs were designated S6030 and S6031. Samples were collected by WHC on April 4, 1996, using the VSS, a truck-based sampling method using a heated probe; and the ISVS without particulate prefiltration.

Sampling devices and controls provided for this job included 11 sorbent trains for water and ammonia (seven sample trains and four field blanks); nine SUMMA™ canisters for permanent gases, TO-12 and volatile organic analytes (six samples and three ambient canisters); and 13 TSTs for organic analytes (seven samples, four field blanks, and two trip blanks). The samples and controls were provided to WHC on April 3, 1996. Exposed samples and controls were returned to PNNL on April 16, 1996. Samples and controls were handled, stored, and transported using chain-of-custody (COC) forms to ensure sample quality was maintained.

Samples and controls were handled and stored as per PNNL technical procedure PNL-TVP-07^(b), and upon return to PNNL, were logged into PNNL Laboratory Record Book 55408. Samples were stored at the VAL under conditions (e.g., ambient, refrigerated) required by technical procedures. Access to the samples was controlled and limited to PNNL staff trained in the application of specific technical procedures to handle samples for the tank vapor characterization project. Analyses were performed in the 300 Area at Hanford. Specific analytical methods are described in the text.

Tank headspace samples were analyzed for

- *water and ammonia* using weight gain for water and ion-specific electrode for ammonia,
- *permanent gases* using gas chromatography/thermal conductivity detection (GC/TCD),

(a) Pacific Northwest National Laboratory is operated for the U. S. Department of Energy by Battelle under Contract DE-AC06-76RLO 1830. The previous name for the laboratory was Pacific Northwest Laboratory (PNL), which is used when previously published documents are cited.

(b) PNL-TVP-07, Rev. 2, December, 1995, *Sample Shipping and Receiving Procedure for PNL Waste Tank Samples*, PNL Technical Procedure, Tank Vapor Project, Pacific Northwest Laboratory, Richland, Washington.

- *total non-methane organic compounds* using cryogenic preconcentration followed by gas chromatography/flame ionization detection (GC/FID), and
- *organic vapors* using cryogenic preconcentration followed by gas chromatography/mass spectrometer (GC/MS) detection.

This report provides summary and detailed analytical information related to the samples and controls. Section 2.0 provides a summary of analytical results. Section 3.0 provides conclusions. Descriptions of samples, analytical methods, quality assurance (QA) and quality control issues, and detailed sample results are provided for each category of samples and analyses in Appendices A, B, C, D, and E. Appendix F contains a listing of all target analytes measured during the analysis of samples from this Tank S-102 comparison study. Appendix G contains the completed COC forms.

2.0 Analytical Results

Samples obtained by WHC from the headspace of Tank S-102 on April 4, 1996 (Sample Jobs S6030 and S6031) were analyzed in the PNNL VAL. Summarized results are described in this section. Details of samples, analyses, and data tables are provided in the appendices.

2.1 Water and Ammonia

The complete results of the water and ammonia analysis of Tank S-102 for the two sampling methods can be found in Appendix A. Table 2.1 presents the mean concentration values for these two analytes. Mean water concentration values ranged from 12.2 mg/L in the ISVS samples to 12.7 mg/L in the VSS samples. Mean NH_3 concentration values ranged from 558 ppmv in the ISVS samples to 564 ppmv in the VSS samples.

Table 2.1. Comparison of Water and Ammonia Mean Values for Samples Collected from the Headspace of Tank S-102 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
Water (mg/L)	12.7	12.2
Ammonia (ppmv)	564	558

2.2 Permanent Gases

The complete results of the permanent gas analyses of Tank S-102 for the two sampling methods can be found in Appendix B. Table 2.2 presents the mean concentration values for the five permanent gases measured. Hydrogen (H_2) and nitrous oxide (N_2O) were measured above the analytical method estimated quantitation limit (EQL). Methane (CH_4), carbon dioxide (CO_2), and carbon monoxide (CO) were not observed in any of the samples. No significant differences were found in the mean concentrations of H_2 and N_2O for the two different sampling methods.

Table 2.2. Comparison of Permanent Gas Mean Values for Samples
Collected from the Headspace of Tank S-102 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
H ₂ (ppmv)	622	632
CO ₂ (ppmv)	4 J	4 J
N ₂ O (ppmv)	646	645
CH ₄ (ppmv)	5 U	5 U
CO (ppmv)	4 U	4 U

2.3 Total Non-Methane Organic Compounds

The complete results of the U.S. Environmental Protection Agency (EPA) TO-12 analyses for TNMOCs in Tank S-102 can be found in Appendix C. A summary of those results can be found in Table 2.3. The TNMOC average concentrations ranged from 15.5 mg/m³ in the VSS SUMMA™ samples to 15.9 mg/m³ in the ISVS samples.

Table 2.3. Comparison of TO-12 Mean Values for Samples Collected from the
Headspace of Tank S-102 Using VSS and ISVS

	<u>VSS</u>	<u>ISVS</u>
TO-12 (mg/m ³)	15.5	15.9

2.4 Organic Compounds from SUMMA™ Canisters

The complete results of the organic vapor analyses from SUMMA™ canisters from Tank S-102 can be found in Appendices D and F. A summary of those results can be found in Table 2.4.

In summary, methanol and ethanol were the most abundant compounds identified in each of the SUMMA™ canister samples. Dodecane and tetradecane were not observed in any samples from Tank S-102. Tributyl phosphate (TBP) was measured as a tentatively identified compound (TIC) but was not found in any of the SUMMA™ canisters measured. Based on the average values for each of the sampling methods, the highest concentrations of methanol, ethanol, propanol, hexane, and 1-butanol were observed in the ISVS samples. The highest concentrations of acetonitrile, acetone, and tetrahydrofuran were observed in the VSS samples. Traces of tridecane were observed in a single SUMMA™ ISVS sample.

2.5 Organic Compounds from Triple Sorbent Traps

The complete results of the organic vapor analyses from TSTs from Tank S-102 can be found in Appendices E and F. A summary of those results can be found in Table 2.5.

In summary, methanol and ethanol were the most abundant compounds identified in each of the trap samples. Tributyl phosphate was not observed in any samples from Tank S-102. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetonitrile, propanol, hexane, and 1-butanol were observed in the VSS samples. The highest concentrations of acetone, tetrahydrofuran, dodecane, tridecane and tetradecane were observed in the ISVS samples.

Table 2.4 Summary of SUMMA™ Sample Results for Samples Collected from the Headspace of Tank S-102 on 4/4/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Average	8633 E,Y	9982 E,Y	34	605	44	34	2.5	436	U	U	U	Z
ST DEV	2277	2301	5.1	229	13	5.0	0.7	89				
% RSD	26	23	15	38	29	15	26	20				
ISVS												
Average	8707 E,Y	10896 E,Y	24	477	46	29	3.2 J	633	U	0.8 J	U	Z
ST DEV	1707	2711	4.5	147	7.2	4.4	0.3	290				
% RSD	20	25	19	31	16	15	8	46				

Data Qualifier Flag

E Target compounds exceeds Upper Quantitation Limit (UQL)

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z TBP was analyzed as a TIC; however, was not identified in the sample.

Table 2.5 Summary of Triple Sorbent Trap Sample Results for Samples Collected from the Headspace of Tank S-102 on 4/4/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Average	4087 E,Y	6246 E,Y	80	230	51	43	U	535	2.0 J	5.8 J	21	<0.83 Z
ST DEV	1400	1139	10	18	5.1	1.2		49	0.3	1.0	5.3	
% RSD	34	18	13	7.6	10	2.7		9.2	15	17	25	
ISVS												
Average	3012 E,Y	4052 E,Y	81	245	44	39	11	407	2.2 J	8.5 J	27 J	<0.88 Z
ST DEV	1113	1157	17	15	6.1	2.1		69	0.2	3.3	13	
% RSD	37	29	21	6.1	14	5.3		17	9.3	39	49	

Data Qualifier Flags

- E Target compound exceeds upper quantification limit (UQL).
- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

3.0 Conclusions

The air concentrations of H_2O and NH_3 , permanent gases, total non-methane organic compounds, and organic vapors were determined from samples from the headspace of Tank S-102 sampled on April 4, 1996. WHC sample job numbers were S6030 and S6031. The gas and vapor concentrations were based either on whole-volume samples (SUMMA™ canisters) or on triple sorbent traps exposed to sample flow. In the case of the canisters, the concentrations were based on analytical results of subsamples obtained directly from the canisters. In the case of the sorbent traps, concentrations were based on analyses by the VAL and sample volumes reported by WHC. Known sampling and analytical variances from established QA requirements, where significant, were documented in this report, as required by the SAP (Homi 1996).

4.0 Reference and Further Reading

Reference

Homi, C.S.- 1996. *Sampling and Analysis Plan for Tank Vapor Sampling Comparison Test*. WHC-SD-WM-TSAP-073, Rev. 1B, Westinghouse Hanford Company, Richland, Washington.

Further Reading

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Appendix A

Tank Vapor Characterization:

Water and Ammonia

Appendix A

Tank Vapor Characterization: Water and Ammonia

Solid sorbent traps, prepared in multi-trap sampling trains, were supplied to Westinghouse Hanford Company (WHC) for sampling the tank headspace using the VSS and ISVS systems. Blanks, spiked blanks (when requested by the SAP), and exposed samples were returned to Pacific Northwest National Laboratory (PNNL) for analysis. Analyses were performed to provide information on the tank headspace concentration of ammonia (NH_3) and water (H_2O). Procedures were similar to those developed previously during sample jobs performed with the VSS connected to the headspace of Tank C-103 (Ligotke et al. 1994). During those sample jobs, control samples provided validation that the sorbent tubes effectively trapped NH_3 and mass. Samples were prepared, handled, and disassembled as described in Technical Procedure PNL-TVP-09^(a). Analytical accuracy was estimated based on procedures used. Sample preparation and analyses were performed following PNNL quality assurance (QA) impact level II requirements.

A.1 Sampling Methodology

Standard glass tubes containing sorbent materials to trap vapors of NH_3 and H_2O (supplied by SKC Inc., Eighty Four, Pennsylvania) were obtained, prepared, and submitted for vapor sampling. The sorbent traps were selected based on their use by the Occupational Safety and Health Administration to perform workplace monitoring and because of available procedures and verification results associated with that particular application. The typical sorbent traps used consisted of a glass tube containing a sorbent material specific to the compound of interest. In general, the tubes contained two sorbent layers, or sections; the first layer was the primary trap, and the second layer provided an indication of breakthrough. In the tubes, sorbent layers are generally held in packed layers separated by glass wool. The sorbent traps, with glass-sealed ends, were received from the vendor.

The type and nominal quantity of sorbent material varied by application. Sorbent traps were selected for the tank sample job and included the following products. The NH_3 sorbent traps contained carbon beads impregnated with sulfuric acid; nominally, 500 mg were contained in the primary and 250 mg in the breakthrough sections. The NH_3 was chemisorbed as ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$. The water traps contained 300 mg of silica gel in the primary and 150 mg in the breakthrough sections.

Sorbent trains provided to trap inorganic compounds included all or some of the following: samples, spiked samples, spares, blanks, and spiked blanks. Sorbent trains were prepared from same-lot batches. After sample preparation, sorbent trains were stored at $\leq 10^\circ\text{C}$ because of handling recommendations for the oxidizer tubes attached to some samples. After receipt of exposed and

(a) Pacific Northwest Laboratory. 12/95. *Sorbent Trap Preparation for Sampling and Analysis: Waste Tank Inorganic Vapor Samples*, PNL-TVP-09 (Rev. 2), PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.

radiologically cleared samples from WHC and disassembly of the sorbent trains, samples were provided to the analytical laboratory at ambient temperature.

The sorbent traps were prepared in multi-trap sorbent trains configured so sample flow passed in order through the traps, targeting specific analytes, and then through a desiccant trap. The specific order of traps within the various sorbent trains is described in Section A.4. The ends of the glass-tube traps were broken, and the traps were weighed and then connected to each other using uniform lengths of 3/8-in. perfluoroalkoxy-grade Teflon® tubing. The tubing was heated in hot air and forced over the open ends of the traps to form a tight seal. The inlets of the sorbent trains each consist of a short section of tubing that has a 3/8-in. stainless steel Swagelok® nut, sealed using a Swagelok® cap. The trailing ends of the sorbent trains (the downstream end of the traps containing silica gel) were each sealed with red plastic end caps provided by the manufacturer. The sorbent-tube trains remained sealed other than during the actual sampling periods. During vapor sampling, C-Flex® tubing was provided by WHC to connect the downstream ends of the sorbent trains to the sampling manifold exhaust connections.

A.1.1 Concentration Calculations. The concentrations of target compounds in the tank headspace were determined from sample results, assuming effective sample transport to the sorbent traps. Concentration, in parts per million by volume (ppmv), was determined by dividing the mass of the compound, in μmol , by the volume of the dried tank air sampled in moles. The micromolar sample mass was determined by dividing the compound mass, in μg , by the molecular weight of the compound, in g/mol. The molar sample volume was determined, excluding water vapor, by dividing the standard sample volume (at 0°C and 760 torr), in L, by 22.4 L/mol. For example, the concentration by volume of a 3.00-L sample containing 75.0 μg of NH_3 is given by

$$\frac{75.0 \mu\text{g}}{17.0 \text{ g/mol}} \left[\frac{3.00 \text{ L}}{22.4 \text{ L/mol}} \right]^{-1} = 32.9 \text{ ppmv} \quad (\text{A.1})$$

This calculational method produces concentration results that are slightly conservative (greater than actual) because the volume of water vapor in the sample stream is neglected. The volume of water vapor is not included in the measured sampled volume because of its removal in desiccant traps upstream of the mass flowmeter. However, the bias is generally expected to be small. For a tank headspace temperature of 35°C, the magnitude of the bias would be about 1 to 6%, assuming tank headspace relative humidities of 20 to 100%, respectively. The concentration of mass (determined gravimetrically) was also per dry-gas volume at standard conditions.

A.2 Analytical Procedures

The compounds of interest were trapped using solid sorbents and chemisorption (adsorption of water vapor). Analytical results were based on extraction and analysis of selected ions. Analytical procedures used are specified in the text.

A.2.1 Ammonia Analysis. The sorbent material from the NH_3 -selective sorbent traps was placed into labeled 20-mL glass scintillation vials. Vials containing front-, or primary-, section sorbent material were treated with 10.0 mL of deionized water (DIW), and vials containing back-up-section sorbent material were treated with 5.0 mL of DIW. After extraction, the NH_3 sorbent traps were analyzed using the selective ion electrode procedure PNL-ALO-226 Rev. 0^(a). Briefly, this method includes 1) preparing a 1000- $\mu\text{g/mL}$ (ppm) NH_3 stock standard solution from dried reagent-grade NH_4Cl and DIW, 2) preparing 0.1-, 0.5-, 1.0-, 10-, and 100-ppm NH_3 working calibration standards by serial dilution of the freshly made stock standard, 3) generating an initial calibration curve from the measured electromotive force signal versus NH_3 concentration data obtained for the set of working standards, 4) performing a calibration-verification check, using a mid-range dilution of a certified National Institute for Standards and Technology (NIST)-traceable 0.1 M NH_4Cl standard from an independent source, after analyzing every five or six samples, 5) continuing this sequence until all samples of the batch have been measured, including duplicates and spiked samples, and 6) remeasuring the complete set of calibration standards (at the end of the session). Electromotive force (volts) signal measurements obtained for samples are compared to those for standards, either graphically or algebraically (using linear regression) to determine NH_3 concentration in the samples.

A.2.2 Mass (Water) Analysis. Sorbent traps used to make each sample train were weighed using a semi-micro mass balance, after labeling and breaking the glass tube ends, without plastic end caps in accordance with procedure PNL-TVP-09. After receipt of exposed samples, the sorbent traps were again weighed to determine the change in mass. Records of the measurements were documented on sample-preparation data sheets. The mass concentration, generally roughly equal to the concentration of water, was determined by dividing the combined change in mass from all traps in a sorbent train by the actual volume of gas sampled. Field blanks were used to correct results.

A.3 Quality Assurance/Quality Control

Analytical work was performed according to quality levels identified in the project QA plan and several PNNL documents including PNL-MA-70 (Part 3), PNL-ALO-212, PNL-ALO-226, and Quality Assurance Plan ETD-002. The samples were analyzed following PNNL Impact Level II. A summary of the analysis procedures and limits for the target inorganic compounds is provided in Table A.1. The table also shows generic expected notification ranges and describes related target analytical precision and accuracy levels for each analyte; the information in the table is based on the data quality objective assessment by Osborne et al. (1995). From the table, it can be seen that the method detection limit (MDL) required to resolve the analyte at one-tenth of the recommended exposure limit for each of the target analytes is achieved using current procedures and with a vapor-sample volume of 2 L and a desorption-solution volume of 3 mL (10 mL for NH_3).

The accuracy of concentration measurements depends on potential errors associated with both sampling and analysis (see Section A.4). Sampling information, including sample volumes, was provided by WHC; sample-volume uncertainty was not provided. The uncertainty of analytical results, which depends on the method used, was estimated to be within allowable tolerances (Osborne

(a) Procedure entitled "Ammonia (Nitrogen) in Aqueous Samples," PNL-ALO-226, in the *Analytical Chemistry Laboratory (ACL) Procedure Compendium*, Vol. 3: Inorganic Instrumental Methods. Pacific Northwest Laboratory, Richland, Washington.

et al. 1995; Table A.1). For NH_3 analyses, the accuracy of laboratory measurements by selective ion electrode was estimated to be $\pm 5\%$ relative, independent of concentration at $1 \mu\text{g/mL}$ or greater levels. The uncertainty includes preparation of standards, purity of the ammonium salt used to prepare standards, potential operator bias, ambient temperature variations, etc. Working standards are traceable to NIST standard reference material by using an independent calibration verification standard certified to be NIST traceable.

Table A.1. Analytical Procedures, Quantitation Limits, and Notification Levels for Selected Inorganic Analytes^(a)

<u>Analyte</u>	<u>Formula</u>	<u>Procedure</u>	EQL ^(b) <u>(μg)</u>	EQL ^(b) <u>(ppmv)</u>	Notification Level ^(c) <u>(ppmv)</u>
Ammonia	NH_3	PNL-ALO-226	1.0	0.7	≥ 150
Mass (water) ^(d)	n/a	PNL-TVP-09	0.6 mg	0.2 mg/L	n/a

(a) Analytical precision and accuracy targets for results in the expected ranges equal $\pm 25\%$ and 70 to 130%, respectively (Osborne et al. 1995).

(b) The lowest calibration standard is defined as the EQL.

(c) As per Table 7-1 in Osborne et al. (1995). Notification levels require verbal and written reports to WHC on completion of preliminary analyses.

(d) The vapor-mass concentration, thought to be largely water vapor, is determined gravimetrically.

n/a = not applicable.

The accuracy of measurements of sample mass is typically $\pm 0.1 \text{ mg}$, or much less than 1% of the mass changes of most samples. The analytical accuracy of measurements of the change in mass of sorbent trains, based on the variability in mass change of field-blank sorbent trains, is determined for each sample job and is typically about $\pm 1 \text{ mg}$ per five-trap sorbent train.

A.4 Water and Ammonia Sample Results

Table A.2 lists results of the water and ammonia analysis from samples collected from the headspace of Tank S-102. These samples were collected through the VSS and through the ISVS systems. A total of 11 samples were collected with the two different sampling methods. The samples were analyzed for ammonia and water on April 25, 1996. Mean water concentration values ranged from 12.2 mg/L in the ISVS samples to 12.7 mg/L in the VSS samples. Mean ammonia concentration values ranged from 558 ppmv in the ISVS samples to 564 ppmv in the VSS samples. Sample S6031-A36.S40 was not analyzed due to the loss (spillage) of the sample in the laboratory.

**Table A.2 Water and Ammonia Analysis Results for Samples Collected
from the Headspace of Tank S-102 on 4/4/96**

VSS Truck Samples	H₂O mg/L	NH₃ ppmv
S6031-A17.S43	12.5	534
S6031-A18.S44	12.7	571
S6031-A19.S45	12.9	576
S6031-A20.S46	12.8	573
Average	12.7	564
% RSD	1.3	3.5

ISVS		
S6030-A34.S38	12.0	576
S6031-A35.S39	12.2	540
S6031-A36.S40	12.5	NA
Average	12.2	558
% RSD	2.1	4.6

NA Sample not analyzed due to sample spillage within the laboratory.

A.5 References

Clauss, T. W., M. W. Ligothke, B. D. McVeety, K. H. Pool, R. B. Lucke, J. S. Fruchter, and S. C. Goheen. 1994. *Vapor Space Characterization of Waste Tank 241-BY-104: Results from Samples Collected on 6/24/94*. PNL-10208. Pacific Northwest Laboratory, Richland, Washington.

Ligothke, M. W., K. H. Pool, and B. D. Lerner. 1994. *Vapor Space Characterization of Waste Tank 241-C-103: Inorganic Results from Sample Job 7B (5/12/94 - 5/25/94)*. PNL-10172, Pacific Northwest Laboratory, Richland, Washington.

Osborne, J. W., J. L. Huckaby, E. R. Hewitt, C. M. Anderson, D. D. Mahlum, B. A. Pulsipher, and J. Y. Young. 1995. *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Resolution*. WHC-SD-WM-DQO-002, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

Appendix B

Tank Vapor Characterization:

Permanent Gases

Appendix B

Tank Vapor Characterization: Permanent Gases

B.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant-free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02^(a). The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis. If the canister is verified as clean by TO-12, the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 μ L of distilled water and labeled with a field-sampling identification. Canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use.

B.2 Analytical Procedure

The SUMMA™ canister samples were analyzed for permanent gases according to PNNL Technical Procedure PNL-TVP-05^(b) with the exceptions listed in the following text and in the quality assurance/quality control section of this report. This method was developed in-house to analyze permanent gases, defined as hydrogen (H_2), carbon dioxide (CO_2), carbon monoxide (CO), methane (CH_4), and nitrous oxide (N_2O), by gas chromatograph/thermal conductivity detection (GC/TCD). Aliquots of sampled air are drawn directly from each canister into a 5-mL gas-tight syringe and injected into a Hewlett-Packard 5890 GC/TCD fitted with a loop injector valve and a column switching valve. An aliquot of 5 mL is used so that the 1.0-mL injection loop is completely purged with sample air, ensuring that no dilution of the sample takes place within the injection loop. One set of GC conditions is used to analyze for CO , CO_2 , N_2O , and CH_4 using Helium (He) as the carrier gas. A second GC analysis is performed for H_2 (using nitrogen as the carrier gas) to enhance the signal sensitivity and lower the detection limit for this analyte. The permanent gases and the derived EQLs are listed in Table B.1.

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- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.
 - (b) Pacific Northwest Laboratory. 12/95. *Analysis Method for the Determination of Permanent Gases in Hanford Waste Tank Vapor Samples Collected in SUMMA™ Passivated Stainless Steel Canisters*, PNL-TVP-05 (Rev. 1). PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.

Table B.1. Analytical Procedures and Detection Limits for Permanent Gases

<u>Analyte</u>	<u>Formula</u>	<u>Procedure</u>	<u>Instrument Detection Limit (ppmv)</u>	<u>Estimated Quantitation Limit (ppmv)</u>
Carbon Dioxide	CO ₂	PNL-TVP-05	2.4	24
Carbon Monoxide	CO	PNL-TVP-05	3.2	32
Methane	CH ₄	PNL-TVP-05	4.3	43
Hydrogen	H ₂	PNL-TVP-05	3.1	31
Nitrous Oxide	N ₂ O	PNL-TVP-05	2.0	20

B.3 Quality Assurance/Quality Control

Standards for the permanent gas analysis were blended from commercially prepared and certified standards for each of the analytes reported in Table B.1. The instrument was calibrated for CO, CO₂, N₂O, and CH₄ over a range of 25 to 2100 parts per million by volume (ppmv) using standards at five different concentrations and He as a carrier gas. A similar procedure was followed for H₂, except the carrier gas was changed to N₂. An average response factor from the calculation was used for quantification of compound peak area.

Each analyte was quantitated by comparison of sample analyte peak area to the calibration plot generated for the compound. An instrument detection limit (IDL) study was conducted and performance data are presented in Table B.1. The EQL for the method has also been established as 10 times the IDL. Before and after each sample analysis set, a gas standard was run to evaluate system performance and to measure system accuracy. The calculated concentration of the individual gases in the standards fell within $\pm 25\%$ of the expected concentrations. One sample was run in duplicate to provide a measure of method precision. Results of the replicate analysis are presented in Table B.2. An N₂ reagent blank, an ambient-air sample collected ~10 m upwind of Tank S-102, and the ambient air collected through the VSS and ISVS were used as method blanks and used to determine the potential for analyte interferences in the samples.

B.4 Permanent Gases Sample Results

Table B.2 lists results of the permanent gas analysis from samples collected from the headspace of Tank S-102 and ambient air collected near Tank S-102. These samples were collected through the VSS and ISVS systems. A total of nine samples were collected with the two different sampling methods. The samples were analyzed on April 17 and 18, 1996. Replicate analyses on SUMMA™ canisters were conducted on two samples within each sampling method set. Hydrogen and nitrous oxide were observed above the EQL in all of the tank headspace samples. Average hydrogen concentrations ranged from 622 ppmv in the VSS samples to 632 ppmv in the ISVS samples. Average nitrous oxide concentrations ranged from 645 ppmv in the ISVS samples to 646 ppmv in the VSS samples. Methane and carbon monoxide concentrations were below the IDL, and carbon dioxide was just above the IDL in several samples..

Table B.2 Permanent Gas Analysis Results for Samples Collected from the Headspace of Tank S-102 and Ambient Air Collected Near Tank S-102 on 4/4/96

VSS Truck Samples	H₂ (ppmv)		CO₂ (ppmv)		N₂O (ppmv)		CH₄ (ppmv)		CO (ppmv)
S6031-A03.025 (Ambient)	3.1	U	343		2.0	U	4.3	U	3.2
S6031-A04.079 (Ambient)	3.1	U	343		2.0	U	4.3	U	3.2
S6031-A06.223	620		5.5	J	644		4.3	U	3.2
S6031-A07.231	632		3.8	J	648		4.3	U	3.2
S6031-A05.148	613		2.4	U	646		4.3	U	3.2
Average	622		4.7	J	646		4.3	U	3.2
% RSD	1.5		NA		0.3		NA		NA
S6031-A07.231 (REP)	584		2.4	U	650		4.3	U	3.2
ISVS									
S6030-A27.265 (Ambient)	3.1	U	347		2.0	U	4.3	U	3.2
S6030-A28.266	637		5.3	J	640		4.3	U	3.2
S6030-A30.269	617		2.4	U	648		4.3	U	3.2
S6030-A29.267	640		2.4	U	647		4.3	U	3.2
Average	632		5.3	J	645		4.3	U	3.2
% RSD	2.0		NA		0.7		NA		NA
S6030-A29.267(REP)	647		2.4	U	648		4.3	U	3.2

NA Not Applicable

Data Qualifier Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Appendix C

Tank Vapor Characterization:

Total Non-Methane Organic Compounds

Appendix C

Tank Vapor Characterization: Total Non-Methane Organic Compounds

C.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant-free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02^(a). The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis. If the canister is verified as clean by TO-12, the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 μ L of distilled water and labeled with a field-sampling identification. Canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use.

C.2 Analytical Procedure

The SUMMA™ canister samples were analyzed according to PNNL Technical Procedure PNL-TVP-08^(b), which is similar to U.S. Environmental Protection Agency (EPA) compendium Method TO-12. The method detection limits in the sub mg/m³ are required to determine total non-methane organic compounds (TNMOC) concentration in the tank samples.

The method uses an EnTech 7000 cryoconcentration system interfaced with a Hewlett-Packard 5890 gas chromatograph/flame ionization detector (GC/FID). The EnTech concentrator is used to pull a metered volume of 50 to 100 mL of sample air from the SUMMA™ canister mounted on an EnTech 7016CA 16-canister autosampler. The sample is cryogenically concentrated, and constituents are trapped in a stainless steel tube containing glass beads and Tenax. The glass bead/Tenax trap is heated to 180°C and purged with ultra high purity (UHP) helium (He). The purged TNMOCs are carried by a UHP He stream to the GC equipped with an FID where gross organic content is detected and measured.

The GC oven is programmed to run at a 150°C isothermal temperature. Chromatographic separation is not needed in this method since quantitation is from the entire FID response over the run time.

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- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.
 - (b) Pacific Northwest Laboratory. 12/95. *Determination of TO-12 Total Nonmethane Organic Compounds in Hanford Waste Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Flame Ionization Detection*, PNL-TVP-08 (Rev. 1), PNL Technical Procedure, Pacific Northwest Laboratory, Richland, Washington.

Twenty-four hours before the analysis, the SUMMA™ canister samples are pressurized with purified air (supplied by Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The starting pressure was first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure. For example, if the canister had a starting pressure of 740 torr, it was pressurized to 1480 torr. The sample dilution was taken into account when calculating the analysis results.

C.3 Quality Assurance/Quality Control

This method requires user calibration (category 2 measuring and test equipment) of the analytical system in accordance with QA plan ETD-002.

The TNMOC is calibrated by using propane as the calibration standard. The instrument calibration mixture for the PNL-TVP-08 analysis consists of National Institute for Standards and Technology (NIST) 99.999% propane analyzed using an average response factor method for calibration.

A continuing calibration verification (CCV) standard of 100 ppmv propane is analyzed to confirm acceptability of instrument performance. The initial calibration is then used to quantify the samples.

Immediately before running the analysis sequence, a leak-check procedure, which includes evacuating the transfer lines and monitoring the pressure, must be performed on the sample manifold tower. The control limits on this test require that the change in pressure is < 1.5 psi, and the absolute pressure after evacuation is < 3 psi for each manifold position specified in the sequence table. If this criterion is not met, it must be corrected before the samples are analyzed.

Before the tank samples were analyzed, a diagnostic check was performed on the GC/FID instrument by running a system cleanliness procedure and an instrument continuing calibration as described in PNL-TVP-08. First, two blank volumes of Aadco purified air were analyzed to check the cleanliness of the system. This demonstrates through the analysis of a zero-air blank that the level of interference is acceptable in the analytical system. The system should be cleaned to 0.1 mg/m³ of TNMOCs. Second, an instrument continuing calibration is run using 100-mL UHP propane analyzed using the response factor as an external standard method followed by one blank volume of Aadco air.

C.3.1 Quantitation Results of Target Analytes. The mg/m³ was derived from the five-point multilevel calibration curve from the propane standard using the following equation:

$$\text{mg/m}^3 = \frac{(\text{ng TNMOC}) \times (\text{dilution factor})}{\text{mL sampled volume}} \quad (\text{C.1})$$

The ng/m³ concentrations are calculated from mg/m³ using the equation:

$$\text{ng/m}^3 \text{ TNMOC} = \frac{(\text{ng TNMOC})}{(\text{mL sampled})} \times \text{Dilution Factor} \times \frac{(\text{mg})}{(1 \times 10^6 \text{ mL})} \times \frac{(1 \times 10^6 \text{ mL})}{(\text{m}^3)} \quad (\text{C.2})$$

C.4 Total Non-Methane Organic Compounds Sample Results

Table C.1 lists results of the TO-12 gas analysis from samples collected from the headspace of Tank S-102 and ambient air collected near Tank S-102. These samples were collected through the VSS and ISVS. A total of nine samples were collected for the two sampling methods. The samples were analyzed on April 19, 1996. Replicate analyses on SUMMA™ canisters were conducted on one sample within each sampling method set. Concentrations in the three ambient air samples ranged from 0.24 mg/m³ to 0.75 mg/m³. Average concentrations in the tank samples ranged from 15.50 mg/m³ in the VSS samples to 15.89 mg/m³ in the ISVS samples.

**Table C.1 TO-12 Analysis Results for Samples Collected from the Headspace
of Tank S-102 and Ambient Air Near Tank S-102 on 4/4/96**

VSS Truck Samples	TO-12 mg/m3	
S6031-A03.025 (Ambient)	0.24	J
S6031-A04.079 (Ambient)	0.41	J
S6031-A06.223	15.63	
S6031-A07.231	15.42	
S6031-A05.148	15.44	
Average	15.50	
% RSD	0.75	
S6031-A07.231 (REP)	15.49	
ISVS		
S6030-A27.265 (Ambient)	0.75	J
S6030-A28.266	15.92	
S6030-A30.269	15.98	
S6030-A29.267	15.77	
Average	15.89	
% RSD	0.68	
S6030-A29.267(REP)	15.92	

Data Qualifier Flags

J Target compound detected above the IDL but below the EQL.

Appendix D

Tank Vapor Characterization:

Organic Compounds from SUMMATM Canisters

Appendix D

Tank Vapor Characterization: Organic Compounds from SUMMA™ Canisters

D.1 Sampling Methodology

Before sending SUMMA™ canisters out to the field for sampling, the canisters are cleaned and verified contaminant free according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-02^(a). The cleaning procedure uses an EnTech 3000 cleaning system that controls 1) filling the canisters with purified humid air and 2) evacuating, for several cycles with applied heat, before allowing the canister to evacuate overnight. The canister is filled a final time with purified humid air for analysis by PNNL Technical Procedure PNL-TVP-03^(b), which is a modification of the U.S. Environmental Protection Agency (EPA) compendium Method TO-14. If the canister is verified as clean, free of TO-14 and unknown contaminants to a level of 5 parts per billion by volume (ppbv), the canister is evacuated to 5 mtorr, tagged, and stored for use in the field. Before sending the canisters out to the field for sampling, the canister vacuum is measured to determine if any leakage has occurred. If the vacuum has remained constant during storage, the canisters are prehumidified with 100 μ L of distilled water and labeled with a field-sampling identification. Cleaned canisters stored more than 30 but less than 60 days are re-evacuated and rehumidified before use. If stored more than 60 days, the canisters are recleaned and validated before use.

D.2 Analytical Procedure

The SUMMA™ canister sample was analyzed according to PNNL Technical Procedure PNL-TVP-03, which is a modified version of EPA compendium Method TO-14. The method uses EnTech 7000 cryoconcentration systems interfaced with a 5972 Hewlett-Packard benchtop gas chromatograph/mass spectrometer (GC/MS). The EnTech concentrator is used to pull a metered volume of sample air from the SUMMA™ canister, cryogenically concentrate the air volume, then transfer the volume to the GC/MS for analysis. A 100-mL volume of sample is measured and analyzed from the tank headspace. The organic components in the sampled air are separated on an analytical column, J&W Scientific DB-1 phase, 60-m by 0.32-mm internal diameter with 3- μ m film thickness. The GC oven is programmed to run a temperature gradient beginning at 40°C, hold for 5 min, and ramp at 4°C per min to a final temperature of 260°C, with a 5-min hold. Twenty-four hours before the analysis, the SUMMA™ canister samples were pressurized with purified air (supplied by Aadco Instruments, Inc., 1920 Sherwood St., Clearwater, Florida 34625). The starting pressure was first measured using a calibrated diaphragm gauge (Cole Parmer), then pressurized to a level exactly twice the original pressure. For example, if the canister had a starting pressure of 740 torr, it

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- (a) Pacific Northwest Laboratory. 8/94. *Cleaning SUMMA™ Canisters and the Validation of the Cleaning Process*, PNL-TVP-02 (Rev. 0), PNL Technical Procedure, Richland, Washington.
- (b) Pacific Northwest Laboratory. 2/96. *Determination of TO-14 Volatile Organic Compounds in Hanford Tank Headspace Samples Using SUMMA™ Passivated Canister Sampling and Gas Chromatographic-Mass Spectrometric Analysis*, PNL-TVP-03 (Rev. 1), PNL Technical Procedure, Richland, Washington.

was pressurized to 1480 torr. This dilution was an effort to improve the precision of the analysis. The sample dilution was taken into account when calculating the analysis results.

The instrument calibration mixture for the PNL-TVP-03 analysis consists of 66 compounds. For this comparison study, only the 12 compounds listed in Table D.1 were considered organic analytes of interest. An initial calibration and CCV were performed for methanol and ethanol. The low level standard (LLS) was used as the EQL for these compounds. Results below the LLS were not reported. It should be noted that these two compounds are not currently part of the operating procedure. Tributyl phosphate was not analyzed as a target compound, but was evaluated as a TIC. The calibration mixture was prepared by blending a commercially prepared TO-14 calibration mixture with a mixture created using a Kin-Tek® permeation-tube standard generation system. The operation of the permeation-tube system follows the method detailed in PNNL Technical Procedure PNL-TVP-06^(a). The standard calibration mix was analyzed using four aliquot sizes ranging from 30 mL to 200 mL, and a response factor for each compound was calculated. The GC/MS response for these compounds has been previously determined to be linearly related to concentration. Instrument detection limits and EQLs have been determined.

Table D.1. Reported Organic Analytes of Interest

Methanol	Acetone
Ethanol	Acetonitrile
1-Butanol	Tetrahydrofuran
Dodecane	Hexane
Tridecane	Propanol
Tetradecane	Tributyl Phosphate (TBP)

D.3 Quality Assurance/Quality Control

Before the tank sample was analyzed, a diagnostic check was performed on the GC/MS instrument by running an instrument "high-sensitivity tune," as described in PNL-TVP-03. Upon satisfactory completion of the instrument diagnostic check, a blank volume of purified nitrogen was analyzed to check the cleanliness of the system. The instrument was then calibrated using a standard gas mixture containing 66 organic compounds. A gas mixture containing bromochloromethane, 1,4-difluorobenzene, chlorobenzene-d₅, and bromofluorobenzene was used as an internal standard (IS) for all blank, calibration standard, and sample analyses. Analyte responses from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. The calibration was generated by calculating the relative response ratios of the IS to calibration standard responses and plotting the ratios against the ratio of the calibration-standard concentration (in ppbv) to the IS concentration. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine

(a) Pacific Northwest Laboratory. 11/94. *Preparation of TO-14 Volatile Organic Compounds Gas Standards*, PNL-TVP-06 (Rev. 0). PNL Technical Procedure, Richland, Washington.

the concentration of target compounds in each sample. Method blanks are analyzed before and after calibration standards and tank headspace samples are analyzed.

D.3.1 Quantitation Results of Target Analytes. The quantitative-analysis results for the target analytes were calculated using the average response factors generated using the IS method described above and in PNL-TVP-03. The conversion from ppbv to mg/m³ assumes standard temperature and pressure (STP) conditions of 760 torr and 273K and was calculated directly from the following equation:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}}{22.4 \text{ L/mol}} \quad (\text{D.1})$$

D.4 Organic Sample Results

Nine SUMMA™ canisters consisting of six samples and three ambient air samples were returned to the laboratory on April 16, 1996, under WHC COC numbers 100061 and 100062. Samples were analyzed on June 27 and 28, 1996.

The results from the GC/MS analysis of the tank headspace SUMMA™ samples are presented in Table D.2. The results of replicate analyses on single SUMMA™ canister samples are presented in Table D.3. The results of the blank sample analyses are presented in Table D.4. Appendix F contains a complete listing of all target analytes measured.

Table D.2 lists the quantitative results for 12 compounds selected for this tank comparison study. Six individual SUMMA™ canister samples were collected using the two different sampling methods. The individual compound values for each of the SUMMA™ canister results for each sampling method were averaged and a standard deviation (ST DEV) and % RSD value calculated. Methanol and ethanol were the most abundant compounds identified in each of the SUMMA™ canister samples. Dodecane, and tetradecane were not observed in any samples from Tank S-102. Tributyl phosphate (TBP) was measured as a tentatively identified compound (TIC) but was not found in any of the SUMMA™ canisters measured. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, propanol, hexane, and 1-butanol were observed in the ISVS samples. The highest concentrations of acetonitrile, acetone, and tetrahydrofuran were observed in the VSS samples. Traces of tridecane were observed in a single SUMMA™ ISVS sample.

Single SUMMA™ canister samples were analyzed in replicate for each of the three different sampling methods. The relative percent differences (RPDs) were calculated and are presented in Table D.3. The RPDs were calculated for analytes detected above the IDL and found in both replicates.

The results of the blank analyses are reported in Table D.4. The only compound consistently observed in the blank samples was acetone. Traces of propanol and 1-butanol were observed in several of the ambient air samples.

The following procedural changes and observations were noted during the analysis of Tank S-102:

Methanol and ethanol are not currently included in the method performance section for System 1; however, both analytes were analyzed by this method. The low level standard is used as the EQL for these compounds. Sample results are flagged with a less-than symbol (<) when less than the EQL value.

The SUMMA™ canister samples were analyzed in two batches. The sample analytical sequence runs (batches) were the following:

Batch #1 (file identifier 16062702 & 3.b) - S6031-A03.025, S6031-A04.079, S6031-A06.223, S6031-A06.223 REP, S6031-A28.266, S6031-A07.231;

Batch #2 (file identifier 16062802 & 3.b) - S6030-A27.265, S6030-A30.269, S6031-A05.148, S6030-A29.267, S6030-A29.267 REP.

Batch #1:

Eight target compounds (dichlorodifluoromethane at 32.3%, acetone at 70.2%, 1,1-dichloroethane at 41.5%, hexane at 47.9%, 1,1,1-trichloroethane at 58.3%, carbon tetrachloride at 57.0%, pyridine at 97.3%, and tetradecane at 42.2%) surpassed the 30% relative standard deviation (% RSD) acceptance criteria for the initial calibration. Acetone fluctuation in the first calibration level may have caused its relative response factor in the CCV sample also to exceed the 25% difference (% D) acceptance criteria. Acetone was detected in tank samples at concentrations between the EQL and the upper quantitation limit (UQL). Dichlorodifluoromethane, pyridine, and hexane were found in tank samples at concentrations between the IDL and the EQL. Tetradecane, 1,1-dichloroethane, carbon tetrachloride, and 1,1,1-trichloroethane were not found in the tank samples.

Four target compounds (1,2-dichloro-1,1,2,2-tetrafluoroethane, acetone, trichlorofluoromethane, and pyridine) were outside the $\pm 25\%$ D acceptance criteria for the CCV sample; however, the CCV passed the procedural criterion requiring $\pm 25\%$ D passage for 85% of all target compounds. Acetone and trichlorofluoromethane were found in tank samples at concentrations above the EQL with the exception of trichlorofluoromethane which was not found in the ambient air sample. Their concentration values may be over estimated because of the failed CCV. Pyridine was detected in tank samples at concentrations between the IDL and EQL. The compound 1,2-dichloro-1,1,2,2-tetrafluoroethane was not found in any tank samples.

The compound 1,2,4-trichlorobenzene was found in the CCB above the EQL, but was not detected above the EQL in any tank samples.

The tank sample internal standard quantification area percent recoveries exceeded the acceptance criterion ($50\% < \text{quantification area} < 200\%$) allowed by procedure PNL-TVP-03, Rev. 1. The changes in the internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity. Target compound results found in these

samples could be affected. This problem will continue until a larger GC/MS system is used in the analysis.

Batch #2:

Eight target compounds (dichlorodifluoromethane at 32.3%, acetone at 70.2%, 1,1-dichloroethane at 41.5%, hexane at 47.9%, 1,1,1-trichloroethane at 58.3%, carbon tetrachloride at 57.0%, pyridine at 97.3%, and tetradecane at 42.2%) surpassed the 30% relative standard deviation (% RSD) acceptance criteria for the initial calibration. Acetone fluctuation in the first calibration level may have caused its relative response factor in the CCV sample to also exceed the 25% difference (% D) acceptance criteria. Acetone was detected in tank samples at concentrations between the EQL and the UQL. Dichlorodifluoromethane, pyridine, and hexane were found in tank samples at concentrations between the IDL and the EQL. However, pyridine was not found in tank sample S6031-A07.231 and hexane was not found in tank samples S6031-A03.025 and S6031-A04.079. Tetradecane, 1,1-dichloroethane, carbon tetrachloride, and 1,1,1-trichloroethane were not found in the tank samples.

Eight target compounds (1,2-dichloro-1,1,2,2-tetrafluoroethane, pentane, 1,1,2-trichloro-1,2,2-trifluoroethane, cis-1,2-dichloroethene, hexane, chloroform, cyclohexane, and pyridine) were outside the $\pm 25\%$ D acceptance criteria for the CCV sample; however, the CCV passed the procedural criterion requiring $\pm 25\%$ D passage for 85% of all target compounds. Pentane was found in tank samples at concentrations above the EQL with the exception of ambient air samples. Its concentration values may be over estimated because of the failed CCV. Hexane and pyridine were determined in tank samples at concentrations between the IDL and EQL. However, pyridine was not found in tank sample S6031-A07.231, and hexane was not found in tank samples S6031-A03.025 and S6031-A04.079. The compounds 1,2-dichloro-1,1,2,2-tetrafluoroethane, 1,1,2-trichloro-1,2,2-trifluoroethane, cis-1,2-dichloroethane, chloroform, and cyclohexane were not found in any tank samples. However, cyclohexane was found in tank sample S6030-A28.266 at concentrations above the EQL and in tank sample S6031-A06.223 at concentrations between the IDL and the EQL. Also, 1,1,2-trichloro-1,2,2-trifluoroethane was found in tank sample S6030-A28.266 at concentrations between the IDL and the EQL.

The tank sample internal standard quantification area percent recoveries exceeded the acceptance criterion (50% to 200%) allowed by procedure PNL-TVP-03, Rev. 1. The changes in the internal standard areas were caused by water induced instrument fatigue. This problem is routinely observed with the HP5972 GC/MS system because of its poor pumping capacity. Target compound results found in these samples could be affected. This problem will continue until a larger GC/MS system is used in the analysis.

Table D.2 SUMMA™ Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 4/4/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)		ACETONE (ppbv)		PROPANOL (ppbv)		TETRAHYDROFURAN (ppbv)		HEXANE (ppbv)		1-BUTANOL (ppbv)		DODECANE (ppbv)		TRIDECANE (ppbv)		TETRADECANE (ppbv)		TBP (ppbv)
A05.148	7303	E,Y	9260	E,Y	28		378		38		35		3.3	J	381		U		U		U		Z
A06.223	11262	E,Y	12557	E,Y	38		836		59		39		2.1	J	539		U		U		U		Z
A07.231	7333	E,Y	8129	E,Y	35		602		36		29		2.2	J	389		U		U		U		Z
Average	8633	E,Y	9982	E,Y	34		605		44		34		3	J	436		U		U		U		Z
ST DEV	2277		2301		5.1		229		13		5.0		0.7		89								
% RSD	26		23		15		38		29		15		26		20								
ISVS																							
A28.266	9059	E,Y	9847	E,Y	19	J	639		42		32		3.0	J	583		U		U		U		Z
A29.267	10211	E,Y	13974	E,Y	24		441		54		24		3.2	J	944		U		U		U		Z
A30.269	6852	E,Y	8866	E,Y	28		352		41		31		3.5	J	371		U		0.8	J	U		Z
Average	8707	E,Y	10896	E,Y	24		477		46		29		3.2	J	633		U		0.8	J	U		Z
ST DEV	1707		2711		4.5		147		7.2		4.4		0.3		290								
% RSD	20		25		19		31		16		15		8		46								

Data Qualifier Flag

- E Target compounds exceeds Upper Quantitation Limit (UQL)
- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- Z TBP was analyzed as a TIC; however, was not identified in the sample.

Table D.3 Replicate Analysis of SUMMA™ Canisters for Samples Collected from the Headspace of Tank S-102 on 4/4/96

VSS Truck Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
A06.223	11262 E,Y	12557 E,Y	38	836	59	39	2.1 J	539	U	U	U	Z
A06.223 REP	7364 E,Y	8167 E,Y	25	623	40	31	2.7 J	494	U	U	U	Z
Relative Percent Difference	42	42	41	29	38	23	25	8.7				
ISVS												
A29.267	10211 E,Y	13974 E,Y	24	441	54	24	3.2 J	944	U	U	U	Z
A29.267 Rep	11595 E,Y	16152 E,Y	42	505	61	30	3.3 J	704	U	U	U	Z
Relative Percent Difference	13	14	55	14	12	22	3.1	29				

Data Qualifier Flags

E Target compound exceeds Upper Quantitation Limit (UQL)

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z TBP was analyzed as a TIC; however, was not identified in the sample.

Table D.4 SUMMA™ Blank Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 4/4/96

Blank Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Upwind Ambient	<77 Y	<53 Y	U	54	5.3 J	U	U	11	U	U	U	Z
Upwind Through VSS	<77 Y	<53 Y	U	30	U	U	U	4.4 J	U	U	U	Z
Upwind Through ISVS	<77 Y	<53 Y	U	14	U	U	1.2 J	2.7 J	U	U	U	Z

Data Qualifier Flags

- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- Z TBP was analyzed as a TIC; however, was not identified in the sample.

Appendix E

Tank Vapor Characterization:

Organic Compounds from Triple Sorbent Traps

Appendix E

Tank Vapor Characterization: Organic Compounds from Triple Sorbent Traps

E.1 Sampling Methodology

Samples are collected on Supelco 300 graphite-based triple sorbent traps (TSTs). Before field deployment, each trap is heated to 380°C under inert gas flow for a minimum of 60 min. Tubes are prepared in batches with each tank sampling job constituting one batch. One tube is selected from each batch and run immediately to verify cleanliness. All remaining tubes in the batch receive equal amounts of three surrogate compounds (hexafluorobenzene, toluene-d8, and bromobenzene-d5). One per batch tube is run immediately to verify successful addition of surrogate spikes to that batch. Tubes are then placed in individually labeled plastic shipping tubes (Supelco TD³), which are sealed with gasketed end caps, thus providing a rugged, headspace-free shipping and storage medium. As a precautionary measure, sample tubes are kept in refrigerated storage before and after sampling.

E.2 Analytical Procedure

The Supelco 300 tubes were analyzed according to Pacific Northwest National Laboratory (PNNL) Technical Procedure PNL-TVP-10^(a), with the exceptions noted in Section E.4. The method employs Supelco Carbotrap™ 300 traps for sample collection and preconcentration. The traps are ground-glass tubes (11.5 cm long X 6 mm OD, 4 mm ID) containing a series of sorbents arranged in order of increasing retentivity. Each trap contains 300 mg of Carbotrap™ C, 200 mg of Carbotrap™ B, and 125 mg of Carbosieve™ S-III. The first two sorbents are deactivated graphite with limited sorption power for less volatile compounds. The final trapping stage, the Carbosieve™ S-III, is a graphitized molecular sieve used to retain the most volatile components, including some permanent gases such as Freon-12. Following sample collection and addition of internal standard (IS), the traps are transferred to a Dynatherm ACEM 900 thermal desorber unit for analysis. The trap on the ACEM 900 is then desorbed by ballistic heating to 350°C with the sample then transferred to a smaller focusing trap. A 10:1 split is used during the transfer with 10% of the sample analyzed and the rest retained for reanalysis. The split sample collected on a second identical Carbotrap™ 300 trap is used for repeat analysis on at least one sample per batch. Since the IS also follows the same path, quantitation may be performed directly on the repeat run without changing the calibration. Following desorption from the Carbotrap™ 300 trap, the analyte is transferred to a long, thin focusing trap filled with the same type of trapping materials as the Carbotrap™ 300 traps and in approximately the same ratios. The purpose of the focusing trap is to provide an interface to a capillary gas chromatograph (GC) column, which may be thermally desorbed at a helium (He) flow rate compatible with the column and mass spectrometry (MS) interface (1.2 mL/min). The focusing trap is

(a) Pacific Northwest Laboratory. 2/96. *Determination of Volatile Organic Compounds in Hanford Waste Tank Headspace Samples Using Triple Sorbent Trap Sampling and Gas Chromatograph-Mass Spectrometer Analysis*, PNL-TVP-10 (Rev. 2), PNL Technical Procedure, Richland, Washington.

ballistically heated to thermally desorb components onto a capillary GC column. The column is subsequently temperature programmed to separate the method analytes, which are then detected by MS.

The instrument calibration mixture for the TST analysis consists of 65 compounds. For this comparison study, only the 12 compounds listed in Table E.1 were considered organic analytes of interest. An initial calibration was performed for methanol and ethanol; however, a CCV was not performed. Therefore, concentrations reported are considered estimated for these compounds. The methanol and ethanol LLS was used as the EQL. Results below the LLS were not reported. The calibration mixture is prepared in common with the mixture used for the SUMMA™ analysis (see Section D.2). The standard calibration mix was analyzed using 4 aliquot sizes ranging from 100 mL to 1200 mL, and a response factor for each compound was calculated. Volumes of standard added to the traps are measured by pressure difference on a SUMMA™ canister of known volume. The GC/MS response for these compounds has been previously determined to be linearly related to concentration. Instrument detection limits and EQLs have been determined with the exception of methanol and ethanol. Tributyl phosphate (TBP) is included in the analysis target list based on a calibration performed on January 5 and 9, 1996. The TBP was introduced onto a series of double sorbent traps as a methanolic solution standard rather than a vapor standard. This served to determine the retention time and verify the mass spectral characteristics of the compound. However, verification of the calibration acceptability was not performed because the compound is not present in the CCV. At present, it is not possible to prepare a gas standard from this material. The calibration information on TBP demonstrated that detectability at 0.8 ppbv (based on 200 mL sample) was possible. Tributyl phosphate was not detected in any samples.

Table E.1. Reported Organic Analytes of Interest

Methanol	<i>Acetone</i>
Ethanol	<i>Acetonitrile</i>
1-Butanol	Tetrahydrofuran
Dodecane	Hexane
Tridecane	Propanol
Tetradecane	Tributyl Phosphate (TBP)

NOTE: Compounds shown in italics have an exceptionally high volatility. They are routinely included in the standard and are quantified, but have a restricted linear dynamic range because of the potential for trap breakthrough.

E.3 Quality Assurance/Quality Control

Before the tank sample was analyzed, a diagnostic check was performed on the GC/MS instrument by running a full auto tune, as described in PNL-TVP-10. Upon satisfactory completion of the instrument diagnostic check, a blank tube was analyzed to check the cleanliness of the system. The instrument was then calibrated using a 300-mL volume of standard gas mixture containing 12 compounds shown in Table E.1. A gas mixture containing difluorobenzene, chlorobenzene-d₅, and 1,4 bromofluorobenzene was used as an IS for all calibration standard and sample analyses. Analyte

responses from sample components, ISs, and standards were obtained from the extracted ion plot from their selected mass ion. A continuing calibration was generated by calculating the relative response ratios of the IS to calibration standard responses and plotting the ratios against the ratio of the calibration-standard concentration (in ppbv) to the IS concentration. Once it is determined that the relative response is linear with increasing concentration, an average response factor is calculated for each target analyte and used to determine the concentration of target compounds in each sample.

E.3.1 Quantitation Results of Target Analytes. The quantitative-analysis results for the target analytes were calculated directly from the calibration curve generated using the IS method described above and in PNL-TVP-10. The conversion from ppbv to mg/m³ assumes STP conditions of 760 torr and 273K and was calculated directly from the following equation:

$$\text{mg/m}^3 = \frac{(\text{ppbv}/1000) \times \text{g mol wt of compound}}{22.4 \text{ L/mol}} \quad (\text{E.1})$$

E.4 Triple Sorbent Trap Volatile Organic Sample Results

Thirteen TSTs consisting of 7 samples, 4 field blanks, and 2 trip blanks were returned to the laboratory on April 16, 1996, under WHC COC numbers 100057 and 100058. The samples were analyzed between April 29 and May 2, 1996.

The results from the GC/MS analysis of the tank headspace TST samples are presented in Table E.2. The results of replicate analyses on TST samples are presented in Table E.3. The results of the blank sample analyses are presented in Table E.4. Appendix F contains a complete listing of the 66 analytes measured.

Table E.2 lists the quantitative results for 12 compounds selected for this tank comparison study. Six individual TST samples were analyzed and reported for the two different sampling methods. The individual compound values for each of the TST samples for each sampling method were averaged and a ST DEV and % RSD value calculated. Methanol and ethanol were the most abundant compounds identified in each of the trap samples. Tributyl phosphate was not observed in any samples from Tank S-102. Based on the average values for each of the sampling methods the highest concentrations of methanol, ethanol, acetonitrile, propanol, hexane, and 1-butanol were observed in the VSS samples. The highest concentrations of acetone, tetrahydrofuran, dodecane, tridecane and tetradecane were observed in the ISVS samples.

One triple sorbent trap sample was analyzed in replicate from the VSS sampling method. The RPDs were calculated and are presented in Table E.3. The RPDs were calculated for analytes detected above the IDL and found in both replicates.

The results of the blank analyses are reported in Table E.4. Low levels of methanol and ethanol (relative to the concentrations observed in the tank samples) were observed in several of the VSS and ISVS blanks samples. Traces of acetonitrile, acetone, and tetradecane were observed in several of the blank samples. Contamination from the tape used to seal the bundles was observed during the first comparison study but was absent in the second comparison study TST samples.

All standards, blanks and samples were analyzed under the protocols of procedure PNL-TVP-10, Rev. 2 with the initial calibration performed on April 8, 1996 and subsequent sample runs quantitated against CCVs at the beginning of each daily batch.

Methanol and ethanol are not currently included in the method performance section of the procedure for System 1; however, both analytes were analyzed by this method. The low level standard is used as the EQL for these compounds. Sample results are flagged with a less-than symbol (<) when less than the EQL.

The TST samples were analyzed in 4 batches. The analytical sequence runs (batches) were as follows:

Batch #1 (file identifier 46042901.d) - S6031-A11.892, S6031-A30.760, S6031-A30.760 REP, S6031-A32.742;

Batch #2 (file identifier 46043001.d) - S6030-A31.740, S6031-A08.888, S6031-A09.889, S6031-A09.889 REP, S6031-A11.829 REANALYSIS;

Batch #3 (file identifier 46050101.d) - S6031-A12.893, S6031-A13.894, S6030-A40.761, S6030-A41.764;

Batch #4 (file identifier 46050201.d) - S6031-A14.895, S6031-A15.896.

The following discussion provides details regarding quality control (QC) criterion failures for each batch.

Batch #1:

The first CCV showed a higher than average failure rate. While this CCV was nominally acceptable according to the procedure, a second CCV was run. The second CCV was satisfactory with the exception of chloromethane (46%), cis-1,2-dichloroethene (28%), carbon tetrachloride (34%), pyridine (28%), tridecane (30%), and tetradecane (79%). The CCV passed the procedural criterion requiring $\pm 25\%$ D passage for 85% of the target compounds. The tank samples contained at the most trace levels (i.e., below the EQL) of all these analytes with the exception of tetradecane, and these values may be over or under estimated. The first run for this batch showed unusually high internal standard recoveries. The problem was traced to an unusually low sample splitter setting which resulted in a larger than normal fraction of the sample being transferred to the focusing trap and GC column thus resulting in higher than normal analytical sensitivity. The problem was corrected at the end of the batch. Two samples were rerun, but because of the low internal standard recovery (and thus reduced sensitivity) that data was not considered useable. Careful examination of the other QC controls including surrogates showed that the original data from this run was fully satisfactory. Replicate analysis data from sample S6031-A30.760 was not usable or reported due to unsatisfactory IS responses and surrogate recoveries.

Batch #2:

The first CCV showed an excessively high deviation from the initial calibration for methanol and ethanol. While this CCV was nominally acceptable according to procedure, a second

CCV was run in an attempt to improve the accuracy of the methanol and ethanol determinations. The second CCV was satisfactory for methanol, ethanol and all other target compounds with the exception of heptane (46%), pyridine (31%), and tetradecane (65%). The CCV passed the procedural criterion requiring $\pm 25\%$ D passage for 85% of the target compounds. The tank samples contained minor amounts of heptane and tetradecane, and these results may be over or under estimated due to the uncertainty in the CCV for these compounds. Due to unacceptable internal standard responses for rerun of sample 46043008.d, the results from the rerun will not be used.

Batch #3:

Two target compounds, pyridine (33%) and tetradecane (56%), were outside the $\pm 25\%$ D acceptance criteria for the CCV. However, the CCV passed the procedural criterion requiring $\pm 25\%$ D passage for 85% of the target compounds.

Batch #4:

Three target compounds, methanol (31%), tridecane (54%), and tetradecane (79%), were outside the $\pm 25\%$ D acceptance criteria for the CCV. However, the CCV passed the procedural criterion requiring $\pm 25\%$ D passage for 85% of the target compounds.

Several observations and comments associated with the data generated are discussed below.

1. Several compounds have consistently tended to exhibit less than average precision as demonstrated through comparison of daily calibrations (CCVs) with the initial calibration. These have included methanol, ethanol, pyridine, and tetradecane. Tridecane has also exhibited marginal behavior for some runs. It is believed that the problem with the first three compounds is associated with their high degree of polarity, which evidently causes problems making reproducible vapor standards extracted from SUMMA™ canisters. Tridecane and tetradecane may have too low volatility to produce reliable vapor standards by using essentially volatile compound handling techniques.
2. Sample volumes for all ISVS tank samples have been corrected to STP from the 21°C calibration used on the sampling cart flowmeters. The correction was included in the reported data to provide seamless compatibility with the VSS data that were collected using a 0°C calibration.
3. Very narrow air spikes occasionally are present in chromatograms from this period. This problem was traced to high frequency air leakage through the diffusion pump o-ring and had been corrected for some time but has again reappeared. It has no known impact on data quality but the spikes do appear as features on the total ion chromatogram.
4. All field samples, including field and trip blanks, contained minor amounts of 1-chloro-1,1-difluoroethane. This compound has appeared persistently in most samples sent to the field in the past but was absent in the previous set of samples from Tank S-102. It is believed to be a fugitive refrigerant. That material is never present in tubes archived for a similar amount of time in the 326 Vapor Lab or 329 Building temporary storage. The origin of the material is unclear, but since it has shown up in trip blanks as well as field blanks, the

most likely candidate is one of the refrigerators used for interim storage in the field part of the COC.

5. Methanol and ethanol are not currently included in procedure PNL-TVP-10; however, both compounds were analyzed per this method. The low level standard is used as the EQL for these compounds. Sample results are flagged with a less-than symbol (<) when less than the EQL value.

6. Tributyl phosphate is included in the analysis target list based on a calibration performed on January 5 and 9, 1996. The TBP was introduced onto a series of double sorbent traps as a methanolic solution standard rather than a vapor standard. This served to determine the retention time and verify the mass spectral characteristics of the compound. However, verification of the calibration acceptability was not performed because the compound is not present in the CCV. At present, it is not possible to prepare a gas standard from this material. The calibration information on TBP demonstrated that detectability at 0.8 ppbv (based on 200 mL sample) was possible. Tributyl phosphate was not detected in the tank samples.

7. Target compounds observed in the VSS and ISVS samples at significant levels (above EQL) included: methanol (above UQL), butane, ethanol (above UQL), acetonitrile, acetone, trichlorofluoromethane, pentane, methylene chloride, propanenitrile, propanol, 2-butanone, hexane, tetrahydrofuran, butanenitrile, benzene, 1-butanol, heptane, 4-methyl-2-pentanone, toluene, octane, tetrachloroethene, p/m xylene, nonane, and tetradecane. Several other target analytes were observed in trace amounts (below EQL). A limited number of TICs were detectable including acetaldehyde, isopropanol, N-nitrosodimethylamine, and pyrazine.

The first VSS field blank contained only methylene chloride at levels in excess of the EQL. No target compounds were observed in the second VSS field blank above the EQL. The second ISVS field blank (FB#4) contained several compounds at levels in excess of the EQL including methanol, trichlorofluoromethane, acetone, methylene chloride, 2-butanone, and toluene. These analytes appear to be a combination of environmental contamination associated with assembly of the sample bundle and passive sampling inside the tank. A similar list of analytes was observed in the first ISVS field blank but at somewhat lower levels, and only toluene was detected above the EQL.

The first VSS trip blank contained no compounds at levels in excess of the EQL. The second VSS trip blank contained only methylene chloride in excess of the EQL. Other compounds detected in at least one of the trip blanks at trace levels (below EQL) included methanol, acetone, methylene chloride, trichlorofluoromethane, 2-butanone, trichloroethene, decane, toluene, and tetradecane.

8. Four VSS and three ISVS TST tubes were sent to the field for sampling. All three of the ISVS samples were successfully analyzed. One of the VSS tubes (S6031-A10-890) was found to be damaged during preanalysis inspection and was not used.

Table E.2 Triple Sorbent Trap Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 4/4/96

VSS Truck Samples	METHANOL (ppbv)		ETHANOL (ppbv)		ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
A08.888	5704	E,Y	7239	E,Y	86	213	47	42	U	572	2.1 J	6.9 J	19	<0.83 Z
A09.889	3284	E,Y	6496	E,Y	68	230	50	42	U	554	1.7 J	5.0 J	17	<0.83 Z
A11.892	3274	E,Y	5002	E,Y	86	248	57	44	12	479	2.3 J	5.6 J	27	<0.83 Z
Average	4087	E,Y	6246	E,Y	80	230	51	43	U	535	2.0 J	5.8 J	21	<0.83 Z
ST DEV	1400		1139		10	18	5.1	1.2		49	0.3	1.0	5.3	
% RSD	34		18		13	7.6	10	2.7		9.2	15	17	25	
ISVS														
A31.740	2726	E,Y	4538	E,Y	70	260	47	40	U	468	2.0 J	5.4 J	12 J	<0.88 Z
A32.742	2070	Y	2731	E,Y	73	230	37	37	11	333	2.3 J	12 J	36	<0.88 Z
A33.760	4241	E,Y	4887	E,Y	101	246	48	41	11	421	2.4 J	8.1 J	34	<0.86 Z
Average	3012	E,Y	4052	E,Y	81	245	44	39	11	407	2.2 J	8.5 J	27	<0.88 Z
ST DEV	1113		1157		17	15	6.1	2.1		69	0.2	3.3	13	
% RSD	37		29		21	6.1	14	5.3		17	9.3	39	49	

Data Qualifier Flags

E Target compound exceeds upper quantification limit (UQL).

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Table E.3 Replicate Analysis of Triple Sorbent Trap Samples Collected from the Headspace of Tank S-102 on 4/4/96

VSS Truck Samples	METHANOL	ETHANOL	ACETONITRILE	ACETONE	PROPANOL	TETRAHYDROFURAN	HEXANE	1-BUTANOL	DODECANE	TRIDECANE	TETRADECANE	TBP
A09.889	3284 E,Y	6496 E,Y	68	230	50	42	U	554	1.7 J	5.0 J	17	<0.83 Z
A09.889 Rep	3265 E,Y	6480 E,Y	101	224	48	42	U	538	1.7 J	4.8 J	18	<0.83 Z
Relative Percent Difference	0.6	0.2	39	2.6	4.1	0.0	U	2.9	0.0	4.1	5.7	<0.83 Z

Data Qualifier Flag

- E Target compound exceeds upper quantification limit (UQL).
- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Table E.4 Triple Sorbent Trap Blank Sample Analysis Results for Samples Collected from the Headspace of Tank S-102 on 4/4/96

Blank Samples	METHANOL (ppbv)	ETHANOL (ppbv)	ACETONITRILE (ppbv)	ACETONE (ppbv)	PROPANOL (ppbv)	TETRAHYDROFURAN (ppbv)	HEXANE (ppbv)	1-BUTANOL (ppbv)	DODECANE (ppbv)	TRIDECANE (ppbv)	TETRADECANE (ppbv)	TBP (ppbv)
Field Blank # 1 VSS	<92 Y	<133 Y	U	16 J	U	U	U	U	U	U	10 J	<0.83 Z
Field Blank # 2 VSS	<92 Y	<133 Y	U	15 J	U	U	U	U	U	U	8.2 J	<0.83 Z
Field Blank #3 ISVS	<92 Y	<133 Y	7.7 J	28 J	U	U	U	U	U	U	6.0 J	<0.83 Z
Field Blank #4 ISVS	269	<133 Y	5.0 J	43	U	U	U	U	U	U	6.1 J	<0.83 Z
Trip Blank #1	<192 Y	<133 Y	U	18 J	U	U	U	U	U	U	15 J	<0.83 Z
Trip Blank #2	<192 U	<133 Y	U	17 J	U	U	U	U	U	U	12 J	<0.83 Z

Data Qualifier Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Appendix F

Tank Vapor Characterization:

Target Analytes Measured

Table F.1. SUMMA™ Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6031-A05.148 (ppbv)	Flag	S6031-A06.223 (ppbv)	Flag	S6031-A07.231 (ppbv)	Flag	Mean	Std. Dev.
DICHLORODIFLUOROMETHANE	75-71-8	1.8	J	2.3	J	2.2	J	2.1	0.3
CHLOROMETHANE	74-87-3	4.8	J	11		9.3	J	8.4	3.2
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U		
METHANOL	67-56-1	7303	E,Y	11262	E,Y	7333	E,Y	8633	2277
VINYL CHLORIDE	75-01-4	1.2	J	1.4	J	0.6	U	1.3	0.1
BUTANE	106-97-8	175		148		136		153	20
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U		
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U		
ETHANOL	64-17-5	9260	E,Y	12557	E,Y	8129	E,Y	9982	2301
ACETONITRILE	75-05-8	28		38		35		34	5.1
ACETONE	67-64-1	378		836		602		605	229
TRICHLOROFLUOROMETHANE	75-69-4	23		38		41		34	9.6
PENTANE	109-66-0	10	J	15		18		14	4.0
1,1-DICHLOROETHENE	75-35-4	0.9	U	0.9	U	0.9	U		
METHYLENE CHLORIDE	75-09-2	2.7	J	2.1	J	2.6	J	2.5	0.3
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.5	U	0.5	U	0.5	U		
PROPANOL	71-23-8	38		59		36		44	13
PROPANENITRILE	107-12-0	5.6	J	6.6	J	4.9	J	5.7	0.9
1,1-DICHLOROETHANE	75-34-3	0.5	U	0.5	U	0.5	U		
2-BUTANONE	78-93-3	43		63		40		49	13
CIS-1,2-DICHLOROETHENE	156-59-2	0.8	U	0.8	U	0.8	U		
HEXANE	110-54-3	3.3	J	2.1	J	2.2	J	2.5	0.7
CHLOROFORM	67-66-3	0.5	U	0.5	U	0.5	U		
TETRAHYDROFURAN	109-99-9	35		39		29		34	5.0
1,2-DICHLOROETHANE	107-06-2	0.6	U	0.6	U	0.6	U		
BUTANENITRILE	109-74-0	6.2	J	1.1	U	8.5	J	7.4	1.6
1,1,1-TRICHLOROETHANE	71-55-6	0.5	U	0.5	U	0.5	U		
1-BUTANOL	71-36-3	381		539		389		436	89
BENZENE	71-43-2	14		9.7		11		12	2.2
CARBON TETRACHLORIDE	56-23-5	0.5	U	0.5	U	0.5	U		
CYCLOHEXANE	110-82-7	1.0	U	2.6	J	1.0	U	2.6	
1,2-DICHLOROPROPANE	78-87-5	0.6	U	0.6	U	0.6	U		
TRICHLOROETHENE	79-01-6	0.4	U	0.4	U	0.4	U		
HEPTANE	142-82-5	7.3		4.6		5.2		5.7	1.4
4-METHYL-2-PENTANONE	108-10-1	9.4		12		6.1	J	9.2	3.0
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.5	U	0.5	U	0.5	U		
PYRIDINE	110-86-1	6.2	J	9.6	J	5.3	U	7.9	2.4
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.4	U	0.4	U	0.4	U		
PENTANENITRILE	110-59-8	1.1	U	1.1	U	1.1	U		
1,1,2-TRICHLOROETHANE	79-00-5	0.5	U	0.5	U	0.5	U		
TOLUENE	108-88-3	107		86		83		92	13
1,2-DIBROMOETHANE	106-93-4	0.6	U	0.6	U	0.6	U		
OCTANE	111-65-9	2.7	J	2.4	J	2.0	J	2.4	0.4
TETRACHLOROETHYLENE	127-18-4	5.5	J	4.4	J	4.3	J	4.7	0.7
CHLOROBENZENE	108-90-7	0.5	U	0.6	J	0.6	J	0.6	0.00
HEXANENITRILE	628-73-9	2.0	J	1.4	J	1.2	U	1.7	0.4
ETHYLBENZENE	100-41-4	0.9	J	0.9	J	0.8	J	0.9	0.1
P/M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U		
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	1.3	U		
STYRENE	100-42-5	0.5	U	0.5	U	0.5	U		
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.6	U	0.6	U	0.6	U		
O-XYLENE	95-47-6	1.6	J	1.6	J	1.4	J	1.5	0.1
NONANE	111-84-2	2.2	J	2.1	J	1.6	J	2.0	0.3

Table F.1. SUMMA™ Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6031-A05.148		S6031-A06.223		S6031-A07.231		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
1-ETHYL-2-METHYL BENZENE	611-14-3	0.3	U	0.3	U	0.3	U		
1,3,5-TRIMETHYLBENZENE	108-67-8	0.3	U	0.3	U	0.3	U		
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	U	0.5	U	0.5	U		
DECANE	124-18-5	1.7	J	2.3	J	1.9	J	2.0	0.3
1,3-DICHLOROBENZENE	541-73-1	0.3	U	0.3	U	0.3	U		
1,4-DICHLOROBENZENE	106-46-7	0.3	U	0.3	U	0.3	U		
1,2-DICHLOROBENZENE	95-50-1	0.3	U	0.3	U	0.3	J	0.3	
UNDECANE	1120-21-4	0.5	J	0.9	J	0.5	J	0.6	0.2
1,2,4-TRICHLOROBENZENE	120-82-1	0.4	U	0.4	U	0.4	U		
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U		
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.4	U	0.4	U	0.4	U		
TRIDECANE	629-50-5	0.7	U	0.7	U	0.7	U		
TETRADECANE	629-59-4	0.5	U	0.5	U	0.5	U		

Data Qualifier Flags

E Target compound exceeds upper quantification limit (UQL).

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Table F.2. Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6031-A08.888		S6031-A09.889		S6031-A11.892		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U	2.5	J		
CHLOROMETHANE	74-87-3	1.4	U	1.4	U	1.4	U		
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.41	U	0.41	U	0.41	U		
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U		
METHANOL	67-56-1	5704	E,Y	3284	E,Y	3274	E,Y	4087	1400
BUTANE	106-97-8	65		62		68		65	3
CHLOROETHANE	75-00-3	1.4	U	1.4	U	1.4	U		
ETHANOL	64-17-5	7239	E,Y	6496	E,Y	5002	E,Y	6245.67	1139
ACETONITRILE	75-05-8	86		68		86		80	10
ACETONE	67-64-1	213		230		248		230	18
TRICHLOROFLUOROMETHANE	75-69-4	29		32		30		30	1.5
PENTANE	109-66-0	16		16		17		16	0.6
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U	0.47	U		
METHYLENE CHLORIDE	75-09-2	199		21	J	286		169	135
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U	0.36	U		
PROPANENITRILE	107-12-0	6.5	J	6.2	J	8.3		7.0	1.1
PROPANOL	71-23-8	47		50		57		51	5.1
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U	0.32	U		
2-BUTANONE	78-93-3	40		36		47		41	5.6
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U	0.58	U		
HEXANE	110-54-3	0.32	U	0.32	U	12			
CHLOROFORM	67-66-3	0.53	U	0.53	U	0.53	U		
TETRAHYDROFURAN	109-99-9	42		42		44		43	1.2
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U	0.25	U		
BUTANENITRILE	109-74-0	0.45	U	0.45	U	6.3	U		
BENZENE	71-43-2	20		19		20		20	0.6
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U	0.44	U		
1-BUTANOL	71-36-3	572		554		479		535	49
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U	0.20	U		
CYCLOHEXANE	110-82-7	1.1	U	1.1	U	1.1	U		
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U	0.38	U		
TRICHLOROETHENE	79-01-6	3.2	J	0.61	U	6.0	J	4.6	
HEPTANE	142-82-5	20		20		12		17	4.6
4-METHYL-2-PENTANONE	108-10-1	13		11		14		13	1.5
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U	0.25	U		
PYRIDINE	110-86-1	11	J	12	J	18	J	14	3.8
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U	0.53	U		
PENTANENITRILE	110-59-8	5.6		0.22	U	0.22	U	5.6	
1,1,2-TRICHLOROETHANE	79-00-5	0.26	U	0.26	U	0.26	U		
TOLUENE	108-88-3	132		130		138		133	4.2
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U	0.28	U		
OCTANE	111-65-9	5.6		5.2	J	6.6		5.8	0.72
TETRACHLOROETHYLENE	127-18-4	8.9		8.9		9.9		9.2	0.58
CHLOROBENZENE	108-90-7	0.22	U	0.22	U	0.54	J		
HEXANENITRILE	628-73-9	0.66	U	0.66	U	0.66	U		
ETHYLBENZENE	100-41-4	1.5	J	1.4	J	1.80	J	1.6	0.21
P/M-XYLENE	106-42-3	5.1	J	4.9	J	6.5		5.5	0.87
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U	2.9	U		
STYRENE	100-42-5	0.33	U	0.33	U	0.41	J		
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U	0.77	U		
O-XYLENE	95-47-6	3.0	J	2.8	J	3.7	J	3.2	0.47
NONANE	111-84-2	3.6		3.4		3.9	J	3.6	0.25
1-ETHYL-2-METHYL BENZENE	611-14-3	0.44	U	0.44	U	0.44	U		

Table.F.2. Triple Sorbent Trap Analysis Results for All Target Analytes for VSS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6031-A08.888		S6031-A09.889		S6031-A11.892		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
1,3,5-TRIMETHYLBENZENE	108-67-8	0.44	U	0.44	U	0.44	U		
1,2,4-TRIMETHYLBENZENE	95-63-6	0.47	U	0.47	U	0.61	J		
DECANE	124-18-5	3.1	J	2.7	J	3.5	J	3.1	0.40
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U	0.48	U		
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U	0.43	U		
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U	0.72	U		
UNDECANE	1120-21-4	1.9	J	1.7	J	2.2	J	1.9	0.25
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U	1.9	U		
DODECANE	112-40-3	2.1	J	1.7	J	2.3	J	2.0	0.31
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U	1.8	U		
TRIDECANE	629-50-5	6.9	J	5.0	J	5.6	J	5.8	0.97
TETRADECANE	629-59-4	19		17		27		21	5.3
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z		

Data Qualifier Flags

E Target compound exceeds upper quantification limit (UQL).

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Table F.3. SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6030-A28.266		S6030-A29.267		S6030-A30.269		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	2.2	J	3.8	J	2.0	J	2.7	1.0
CHLOROMETHANE	74-87-3	11		7.5	J	6.2	J	8.2	2.5
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U		
METHANOL	67-56-1	9059	E,Y	10211	E,Y	6852	E,Y	8707	1707
VINYL CHLORIDE	75-01-4	0.6	U	3.9	J	1.6	J	2.8	1.6
BUTANE	106-97-8	166		265		144		192	64
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U		
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U		
ETHANOL	64-17-5	9847	E,Y	13974	E,Y	8866	E,Y	10896	2711
ACETONITRILE	75-05-8	19	J	24		28		24	4.5
ACETONE	67-64-1	639		441		352		477	147
TRICHLOROFLUOROMETHANE	75-69-4	49		38		20		36	15
PENTANE	109-66-0	18		19		12	J	16	3.8
1,1-DICHLOROETHENE	75-35-4	0.9	U	0.9	U	0.9	U		
METHYLENE CHLORIDE	75-09-2	2.9	J	3.5	J	2.6	J	3.0	0.5
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	3.9	J	5.3		3.3	J	4.2	1.0
PROPANOL	71-23-8	42		54		41		46	7.2
PROPANENITRILE	107-12-0	4.3	J	5.0	J	5.6	J	5.0	0.7
1,1-DICHLOROETHANE	75-34-3	0.5	U	0.5	U	0.5	U		
2-BUTANONE	78-93-3	43		45		46		45	1.5
CIS-1,2-DICHLOROETHENE	156-59-2	0.8	U	0.8	U	0.8	U		
HEXANE	110-54-3	3.0	J	3.2	J	3.5	J	3.2	0.3
CHLOROFORM	67-66-3	0.5	U	0.5	U	0.5	U		
TETRAHYDROFURAN	109-99-9	32		24		31		29	4.4
1,2-DICHLOROETHANE	107-06-2	0.6	U	0.6	U	0.6	U		
BUTANENITRILE	109-74-0	3.7	J	1.1	U	3.7	J	3.7	0.00
1,1,1-TRICHLOROETHANE	71-55-6	0.5	U	0.5	U	0.5	U		
1-BUTANOL	71-36-3	583		944		371		633	290
BENZENE	71-43-2	15		11		12		13	2.1
CARBON TETRACHLORIDE	56-23-5	0.5	U	0.5	U	0.5	U		
CYCLOHEXANE	110-82-7	24		1.0	U	24		24	0.00
1,2-DICHLOROPROPANE	78-87-5	0.6	U	0.6	U	0.6	U		
TRICHLOROETHENE	79-01-6	0.4	U	0.4	U	0.4	U		
HEPTANE	142-82-5	6.5		3.4	J	6.4		5.4	1.8
4-METHYL-2-PENTANONE	108-10-1	8.2		15		9.5		11	3.6
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.5	U	0.5	U	0.5	U		
PYRIDINE	110-86-1	6.7	J	12	J	6.5	J	8.4	3.1
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.4	U	0.4	U	0.4	U		
PENTANENITRILE	110-59-8	1.1	U	1.1	U	1.1	U		
1,1,2-TRICHLOROETHANE	79-00-5	0.5	U	0.5	U	0.5	U		
TOLUENE	108-88-3	94		58		91		81	20
1,2-DIBROMOETHANE	106-93-4	0.6	U	0.6	U	0.6	U		
OCTANE	111-65-9	2.0	J	1.4	J	2.5	J	2.0	0.6
TETRACHLOROETHYLENE	127-18-4	4.8	J	3.5	J	4.7	J	4.3	0.7
CHLOROBENZENE	108-90-7	0.5	U	0.5	U	0.6	J	0.6	
HEXANENITRILE	628-73-9	1.2	U	1.9	J	1.3	J	1.6	0.4
ETHYLBENZENE	100-41-4	0.8	J	0.8	J	1.0	J	0.9	0.1
P/M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U		
CYCLOHEXANONE	108-94-1	1.3	U	1.7	J	1.5	J	1.6	0.1
STYRENE	100-42-5	0.5	U	0.5	U	0.5	U		
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.6	U	0.6	U	0.6	U		
O-XYLENE	95-47-6	1.1	J	1.3	J	1.7	J	1.4	0.3
NONANE	111-84-2	1.4	J	1.5	J	1.8	J	1.6	0.2

Table F.3. SUMMA™ Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6030-A28.266		S6030-A29.267		S6030-A30.269		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
1-ETHYL-2-METHYL BENZENE	611-14-3	0.3	U	0.3	U	0.3	U		
1,3,5-TRIMETHYLBENZENE	108-67-8	0.3	U	0.3	U	0.3	U		
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	U	0.5	U	0.5	U		
DECANE	124-18-5	1.9	J	2.2	J	2.0	J	2.0	0.2
1,3-DICHLOROBENZENE	541-73-1	0.3	U	0.3	J	0.3	J	0.3	0.00
1,4-DICHLOROBENZENE	106-46-7	0.3	U	0.3	U	0.3	J	0.3	
1,2-DICHLOROBENZENE	95-50-1	0.3	U	0.4	J	0.4	J	0.4	0.00
UNDECANE	1120-21-4	0.8	J	0.9	J	1.0	J	0.9	0.1
1,2,4-TRICHLOROBENZENE	120-82-1	0.4	U	0.4	U	0.4	U		
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U		
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.4	U	0.4	U	0.4	U		
TRIDECANE	629-50-5	0.7	U	0.7	U	0.8	J	0.8	
TETRADECANE	629-59-4	0.5	U	0.5	U	0.5	U		

Data Qualifier Flags

E Target compound exceeds upper quantification limit (UQL).

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Table F.4. TST Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6030-A31.740		S6030-A32.742		S6030-A33.760		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
DICHLORODIFLUOROMETHANE	75-71-8	0.83	U	1.8	J	5.5	J	3.7	2.6
CHLOROMETHANE	74-87-3	1.5	U	1.5	U	1.4	U		
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.44	U	0.44	U	0.43	U		
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U		
METHANOL	67-56-1	2726	E,Y	2070	E,Y	4241	E,Y	3012	1113
BUTANE	106-97-8	64		58		62		61	3.1
CHLOROETHANE	75-00-3	1.5	U	1.5	U	4.5	J	4.5	
ETHANOL	64-17-5	4538	E,Y	2731	E,Y	4887	E,Y	4052	1157
ACETONITRILE	75-05-8	70		73		101		81	17
ACETONE	67-64-1	260		230		246		245	15
TRICHLOROFLUOROMETHANE	75-69-4	34		30		49		38	10
PENTANE	109-66-0	16		14		15		15	1.0
1,1-DICHLOROETHENE	75-35-4	0.50	U	0.50	U	0.49	U		
METHYLENE CHLORIDE	75-09-2	5.7	J	12	J	25	J	14	9.8
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.38	U	0.59	J	0.67	J	0.63	0.06
PROPANENITRILE	107-12-0	6.6	J	6.6	J	7.2	J	6.8	0.35
PROPANOL	71-23-8	47		37		48		44	6.1
1,1-DICHLOROETHANE	75-34-3	0.34	U	0.34	U	0.33	U		
2-BUTANONE	78-93-3	39		45		50		45	5.5
CIS-1,2-DICHLOROETHENE	156-59-2	0.61	U	0.61	U	0.60	U		
HEXANE	110-54-3	0.34	U	11		11		11	0.00
CHLOROFORM	67-66-3	0.57	U	0.57	U	0.55	U		
TETRAHYDROFURAN	109-99-9	40		37		41		39	2.1
1,2-DICHLOROETHANE	107-06-2	0.26	U	0.26	U	0.26	U		
BUTANENITRILE	109-74-0	0.48	U	4.8	J	5.4		5.1	0.42
BENZENE	71-43-2	19		17		19		18	1.2
1,1,1-TRICHLOROETHANE	71-55-6	0.47	U	0.47	U	0.46	U		
1-BUTANOL	71-36-3	468		333		421		407	69
CARBON TETRACHLORIDE	56-23-5	0.22	U	0.22	U	0.21	U		
CYCLOHEXANE	110-82-7	1.2	U	1.2	U	1.1	U		
1,2-DICHLOROPROPANE	78-87-5	0.40	U	0.40	U	0.39	U		
TRICHLOROETHENE	79-01-6	0.65	U	0.65	U	0.63	U		
HEPTANE	142-82-5	29		17		17		21	6.9
4-METHYL-2-PENTANONE	108-10-1	10		11		12		11	1.0
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.27	U	0.27	U	0.26	U		
PYRIDINE	110-86-1	12	J	19	J	25	J	19	6.5
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.57	U	0.57	U	0.55	U		
PENTANENITRILE	110-59-8	0.23	U	0.62	J	0.22	U	0.62	
1,1,2-TRICHLOROETHANE	79-00-5	0.28	U	0.28	U	0.27	U		
TOLUENE	108-88-3	148		143		150		147	3.6
1,2-DIBROMOETHANE	106-93-4	0.30	U	0.30	U	0.29	U		
OCTANE	111-65-9	4.4	J	4.9	J	5.3	J	4.9	0.45
TETRACHLOROETHYLENE	127-18-4	5.6		6.8		6.9		6.4	0.72
CHLOROBENZENE	108-90-7	0.23	U	0.47	J	0.22	U	0.47	
HEXANENITRILE	628-73-9	0.70	U	0.70	U	0.68	U		
ETHYLBENZENE	100-41-4	5.5		5.3		5.5		5.4	0.12
P/M-XYLENE	106-42-3	21		22		23		22	1.0
CYCLOHEXANONE	108-94-1	3.0	U	3.0	U	2.9	U		
STYRENE	100-42-5	2.3	J	2.4	J	2.7	J	2.5	0.21
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.82	U	0.82	U	0.80	U		
O-XYLENE	95-47-6	7.5		7.6		8.3		7.8	0.44
NONANE	111-84-2	3.0		2.9		3.2		3.0	0.15
1-ETHYL-2-METHYL BENZENE	611-14-3	1.7	J	1.7	J	1.9	J	1.8	0.12

Table F.4. TST Analysis Results for All Target Analytes for ISVS Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6030-A31.740		S6030-A32.742		S6030-A33.760		Mean	Std. Dev.
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag		
1,3,5-TRIMETHYLBENZENE	108-67-8	1.6	J	1.6	J	2.0	J	1.7	0.23
1,2,4-TRIMETHYLBENZENE	95-63-6	4.8	J	4.8	J	5.6		5.1	0.46
DECANE	124-18-5	4.2	J	5.5		5.8		5.2	0.85
1,3-DICHLOROBENZENE	541-73-1	0.51	U	0.84	J	0.50	U	0.84	
1,4-DICHLOROBENZENE	106-46-7	0.88	J	0.84	J	0.87	J	0.86	0.02
1,2-DICHLOROBENZENE	95-50-1	0.76	U	0.76	U	0.74	U		
UNDECANE	1120-21-4	3.5	J	2.8	J	3.0	J	3.1	0.36
1,2,4-TRICHLOROBENZENE	120-82-1	2.0	U	2.0	U	2.0	U		
DODECANE	112-40-3	2.0	J	2.3	J	2.4	J	2.2	0.21
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.9	U	1.9	U	1.8	U		
TRIDECANE	629-50-5	5.4	J	12	J	8.1	J	8.5	3.3
TETRADECANE	629-59-4	12	J	36		34		27	13
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z		

Data Qualifier Flags

E Target compound exceeds upper quantification limit (UQL).

J Target compound detected above the IDL, but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Table F.5. SUMMA™ Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	VSS				ISVS			
		S6031-A06.223		S6030-A29.267					
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	2.3	J	2.3	J	3.8	J	3.7	J
CHLOROMETHANE	74-87-3	11		8.0	J	7.5	J	11	
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U	1.7	U
VINYL CHLORIDE	75-01-4	1.4	J	1.8	J	3.9	J	2.1	J
METHANOL	67-56-1	11262	E,Y	7364	E,Y	10211	E,Y	11595	E,Y
BUTANE	106-97-8	148		151		265		260	
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U	1.2	U
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U	1.9	U
ETHANOL	64-17-5	12557	E,Y	8167	E,Y	13974	E,Y	16152	E,Y
ACETONITRILE	75-05-8	38		25		24		42	
ACETONE	67-64-1	836		623		441		505	
TRICHLOROFLUOROMETHANE	75-69-4	38		48		38		34	
PENTANE	109-66-0	15		25		19		18	
1,1-DICHLOROETHENE	75-35-4	0.9	U	0.9	U	0.9	U	0.9	U
METHYLENE CHLORIDE	75-09-2	2.1	J	3.2	J	3.5	J	3.0	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.5	U	0.5	U	5.3		5.1	
PROPANENITRILE	107-12-0	6.6	J	4.4	J	5.0	J	5.4	J
PROPANOL	71-23-8	59		40		54		61	
1,1-DICHLOROETHANE	75-34-3	0.5	U	0.5	U	0.5	U	0.5	U
2-BUTANONE	78-93-3	63		41		45		48	
CIS-1,2-DICHLOROETHENE	156-59-2	0.8	U	0.8	U	0.8	U	0.8	U
HEXANE	110-54-3	2.1	J	2.7	J	3.2	J	3.3	J
CHLOROFORM	67-66-3	0.5	U	0.5	U	0.5	U	0.5	U
TETRAHYDROFURAN	109-99-9	39		31		24		30	
1,2-DICHLOROETHANE	107-06-2	0.6	U	0.6	U	0.6	U	0.6	U
BUTANENITRILE	109-74-0	1.1	U	1.1	U	1.1	U	7.4	J
1,1,1-TRICHLOROETHANE	71-55-6	0.5	U	0.5	U	0.5	U	0.5	U
1-BUTANOL	71-36-3	539		494		944		704	
BENZENE	71-43-2	9.7		14		11		9.4	
CARBON TETRACHLORIDE	56-23-5	0.5	U	0.5	U	0.5	U	0.5	U
CYCLOHEXANE	110-82-7	2.6	J	1.0	U	1.0	U	1.0	U
1,2-DICHLOROPROPANE	78-87-5	0.6	U	0.6	U	0.6	U	0.6	U
TRICHLOROETHENE	79-01-6	0.4	U	0.4	U	0.4	U	0.4	U
HEPTANE	142-82-5	4.6		7.7		3.4	J	1.8	J
4-METHYL-2-PENTANONE	108-10-1	12		7.0	J	15		9.0	
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.5	U	0.5	U	0.5	U	0.5	U
PYRIDINE	110-86-1	9.6	J	6.6	J	12	J	8.0	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.4	U	0.4	U	0.4	U	0.4	U
PENTANENITRILE	110-59-8	1.1	U	1.1	U	1.1	U	1.1	U
1,1,2-TRICHLOROETHANE	79-00-5	0.5	U	0.5	U	0.5	U	0.5	U
TOLUENE	108-88-3	86		98		58		44	
1,2-DIBROMOETHANE	106-93-4	0.6	U	0.6	U	0.6	U	0.6	U
OCTANE	111-65-9	2.4	J	2.6	J	1.4	J	0.8	J
TETRACHLOROETHYLENE	127-18-4	4.4	J	5.7	J	3.5	J	2.4	J
CHLOROBENZENE	108-90-7	0.6	J	0.6	J	0.5	U	0.5	U
HEXANENITRILE	628-73-9	1.4	J	1.2	U	1.9	J	1.8	J
ETHYLBENZENE	100-41-4	0.9	J	0.9	J	0.8	J	0.6	U
P/M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U	2.7	U
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	1.7	J	1.4	J
STYRENE	100-42-5	0.5	U	0.5	U	0.5	U	0.5	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.6	U	0.6	U	0.6	U	0.6	U
O-XYLENE	95-47-6	1.6	J	1.4	J	1.3	J	1.0	J

Table F.5. SUMMA™ Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	VSS				ISVS			
		S6031-A06,223				S6030-A29,267			
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
NONANE	111-84-2	2.1	J	1.6	J	1.5	J	1.3	J
1-ETHYL-2-METHYL BENZENE	611-14-3	0.3	U	0.3	U	0.3	U	0.3	U
1,3,5-TRIMETHYLBENZENE	108-67-8	0.3	U	0.3	U	0.3	U	0.3	U
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	U	0.5	U	0.5	U	0.5	U
DECANE	124-18-5	2.3	J	1.8	J	2.2	J	2.2	J
1,3-DICHLOROBENZENE	541-73-1	0.3	U	0.3	U	0.3	J	0.3	U
1,4-DICHLOROBENZENE	106-46-7	0.3	U	0.3	U	0.3	U	0.3	U
1,2-DICHLOROBENZENE	95-50-1	0.3	U	0.3	J	0.4	J	0.3	J
UNDECANE	1120-21-4	0.9	J	0.5	J	0.9	J	0.8	J
1,2,4-TRICHLOROBENZENE	120-82-1	0.4	U	0.4	U	0.4	U	0.4	U
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U	1.3	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.4	U	0.4	U	0.4	U	0.4	U
TRIDECANE	629-50-5	0.7	U	0.7	U	0.7	U	0.7	U
TETRADECANE	629-59-4	0.5	U	0.5	U	0.5	U	0.5	U

Data Qualifier Flags

E Target compound exceeds upper quantification limit (UQL).

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Table F.6. Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	(ppbv)	VSS		
			S6031-A09.889		
			Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	0.79	U	0.79	U
CHLOROMETHANE	74-87-3	1.4	U	1.4	U
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.41	U	0.41	U
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U
METHANOL	67-56-1	3284	E,Y	3265	E,Y
BUTANE	106-97-8	62		64	
CHLOROETHANE	75-00-3	1.4	U	1.4	U
ETHANOL	64-17-5	6496	E,Y	6480	E,Y
ACETONITRILE	75-05-8	68		101	
ACETONE	67-64-1	230		224	
TRICHLOROFLUOROMETHANE	75-69-4	32		33	
PENTANE	109-66-0	16		15	
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U
METHYLENE CHLORIDE	75-09-2	21	J	21	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U
PROPANENITRILE	107-12-0	6.2	J	6.2	J
PROPANOL	71-23-8	50		48	
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U
2-BUTANONE	78-93-3	36		33	
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U
HEXANE	110-54-3	0.32	U	0.32	U
CHLOROFORM	67-66-3	0.53	U	0.53	U
TETRAHYDROFURAN	109-99-9	42		42	
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U
BUTANENITRILE	109-74-0	0.45	U	0.45	U
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U
1-BUTANOL	71-36-3	554		538	
BENZENE	71-43-2	19		20	
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U
CYCLOHEXANE	110-82-7	1.1	U	1.1	U
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U
TRICHLOROETHENE	79-01-6	0.61	U	0.61	U
HEPTANE	142-82-5	20		19	
4-METHYL-2-PENTANONE	108-10-1	11		12	
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U
PYRIDINE	110-86-1	12	J	11	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U
PENTANENITRILE	110-59-8	0.22	U	0.22	U
1,1,2-TRICHLOROETHANE	79-00-5	0.26	U	0.26	U
TOLUENE	108-88-3	130		131	
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U
OCTANE	111-65-9	5.2	J	5.3	
TETRACHLOROETHYLENE	127-18-4	8.9		8.6	
CHLOROBENZENE	108-90-7	0.22	U	0.22	U
HEXANENITRILE	628-73-9	0.66	U	0.66	U
ETHYLBENZENE	100-41-4	1.4	J	1.4	J
P/M-XYLENE	106-42-3	4.9	J	5.0	J
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U
STYRENE	100-42-5	0.33	U	0.33	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U
O-XYLENE	95-47-6	2.8	J	2.8	J
NONANE	111-84-2	3.4		3.4	

Table F.6. Triple Sorbent Trap Replicate Analysis Results for All Target Analytes for Samples Collected from the Headspace of Tank S-102 on 4/4/96

Target Analytes	CAS No.	(ppbv)	Flag	VSS	
				S6031-A09.889	
				(ppbv)	Flag
1-ETHYL-2-METHYL BENZENE	611-14-3	0.44	U	0.44	U
1,3,5-TRIMETHYLBENZENE	108-67-8	0.44	U	0.44	U
1,2,4-TRIMETHYLBENZENE	95-63-6	0.47	U	0.47	U
DECANE	124-18-5	2.7	J	3.1	J
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U
UNDECANE	1120-21-4	1.7	J	1.7	J
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U
DODECANE	112-40-3	1.7	J	1.7	J
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U
TRIDECANE	629-50-5	5.0	J	4.8	J
TETRADECANE	629-59-4	17		18	
TBP	126-73-8	<0.83	Z	<0.83	Z

Data Qualifier Flags

- E Target compound exceeds upper quantification limit (UQL).
- J Target compound detected above the IDL but below the EQL.
- U Target compound not detected at or above the IDL.
- Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.
- Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Table F.7. SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6030-A27.265		S6031-A03.025		S6031-A04.079	
		Amb. Air ISVS		Upwind Amb VSS		Amb. thru VSS	
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
DICHLORODIFLUOROMETHANE	75-71-8	1.0	J	1.2	J	1.2	J
CHLOROMETHANE	74-87-3	1.1	U	1.1	U	1.1	U
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	1.7	U	1.7	U	1.7	U
METHANOL	67-56-1	<77	Y	<77	Y	<77	Y
VINYL CHLORIDE	75-01-4	0.6	U	0.6	U	0.6	U
BUTANE	106-97-8	0.9	U	0.9	U	0.9	U
BROMOMETHANE	74-83-9	1.2	U	1.2	U	1.2	U
CHLOROETHANE	75-00-3	1.9	U	1.9	U	1.9	U
ETHANOL	64-17-5	<53	Y	<53	Y	<53	Y
ACETONITRILE	75-05-8	2.2	U	2.2	U	2.2	U
ACETONE	67-64-1	14		54		30	
TRICHLOROFLUOROMETHANE	75-69-4	0.8	U	0.8	U	1.3	J
PENTANE	109-66-0	1.2	U	1.2	U	1.2	U
1,1-DICHLOROETHENE	75-35-4	0.9	U	0.9	U	0.9	U
METHYLENE CHLORIDE	75-09-2	2.9	J	4.7	J	3.1	J
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	10		0.5	U	0.5	U
PROPANOL	71-23-8	1.0	U	5.3	J	1.0	U
PROPANENITRILE	107-12-0	1.6	U	2.2	J	1.6	U
1,1-DICHLOROETHANE	75-34-3	0.5	U	0.5	U	0.5	U
2-BUTANONE	78-93-3	4.8	J	6.9	J	5.0	J
CIS-1,2-DICHLOROETHENE	156-59-2	0.8	U	0.8	U	0.8	U
HEXANE	110-54-3	1.2	J	0.8	U	0.8	U
CHLOROFORM	67-66-3	0.5	U	0.5	U	0.5	U
TETRAHYDROFURAN	109-99-9	0.5	U	0.5	U	0.5	U
1,2-DICHLOROETHANE	107-06-2	0.6	U	0.6	U	0.6	U
BUTANENITRILE	109-74-0	1.1	U	2.5	J	1.4	J
1,1,1-TRICHLOROETHANE	71-55-6	0.5	U	0.5	U	0.5	U
1-BUTANOL	71-36-3	2.7	J	11		4.4	J
BENZENE	71-43-2	1.0	J	1.4	J	1.1	J
CARBON TETRACHLORIDE	56-23-5	0.5	U	0.5	U	0.5	U
CYCLOHEXANE	110-82-7	1.0	U	1.0	U	1.0	U
1,2-DICHLOROPROPANE	78-87-5	0.6	U	0.6	U	0.6	U
TRICHLOROETHENE	79-01-6	0.4	U	0.4	U	0.4	U
HEPTANE	142-82-5	0.4	U	0.4	U	0.4	U
4-METHYL-2-PENTANONE	108-10-1	0.8	U	3.9	J	1.5	J
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.5	U	0.5	U	0.5	U
PYRIDINE	110-86-1	5.8	J	26	J	8.5	J
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.4	U	0.4	U	0.4	U
PENTANENITRILE	110-59-8	1.1	U	2.8	J	1.1	U
1,1,2-TRICHLOROETHANE	79-00-5	0.5	U	0.5	U	0.5	U
TOLUENE	108-88-3	0.5	U	0.6	J	0.5	U
1,2-DIBROMOETHANE	106-93-4	0.6	U	0.6	U	0.6	U
OCTANE	111-65-9	0.5	U	0.5	U	0.5	U
TETRACHLOROETHYLENE	127-18-4	0.6	U	0.6	U	0.6	U
CHLOROBENZENE	108-90-7	0.5	U	0.5	U	0.5	U
HEXANENITRILE	628-73-9	1.2	U	1.2	U	1.2	U
ETHYLBENZENE	100-41-4	0.6	U	0.6	U	0.6	U
P/M-XYLENE	106-42-3	2.7	U	2.7	U	2.7	U
CYCLOHEXANONE	108-94-1	1.3	U	1.3	U	1.3	U
STYRENE	100-42-5	0.5	U	0.5	U	0.5	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.6	U	0.6	U	0.6	U
O-XYLENE	95-47-6	0.6	U	0.6	U	0.6	U

Table F.7. SUMMA™ Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6030-A27.265		S6031-A03.025		S6031-A04.079	
		Amb. Air ISVS	Flag	Upwind Amb VSS	Flag	Amb. thru VSS	Flag
		(ppbv)		(ppbv)		(ppbv)	
NONANE	111-84-2	0.3	U	0.3	U	0.3	U
1-ETHYL-2-METHYL BENZENE	611-14-3	0.3	U	0.3	U	0.3	U
1,3,5-TRIMETHYLBENZENE	108-67-8	0.3	U	0.3	U	0.3	U
1,2,4-TRIMETHYLBENZENE	95-63-6	0.5	U	0.5	U	0.5	U
DECANE	124-18-5	0.5	U	0.5	J	0.6	J
1,3-DICHLOROBENZENE	541-73-1	0.3	U	0.3	U	0.3	U
1,4-DICHLOROBENZENE	106-46-7	0.3	U	0.3	U	0.3	U
1,2-DICHLOROBENZENE	95-50-1	0.3	J	0.3	U	0.3	J
UNDECANE	1120-21-4	0.5	J	0.4	U	0.4	U
1,2,4-TRICHLOROBENZENE	120-82-1	0.7	J	0.4	U	0.4	U
DODECANE	112-40-3	1.3	U	1.3	U	1.3	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.4	U	0.4	U	0.4	U
TRIDECANE	629-50-5	0.7	U	0.7	U	0.7	U
TETRADECANE	629-59-4	0.5	U	0.5	U	0.5	U

Data Qualifier Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Table F.8. Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6031-A12.893		S6031-A13.894		S6030-A40.761		S6030-A41.764		S6031-A14.895		S6031-A15.896	
		VSS FB#1	Flag	VSS FB#2	Flag	ISVS FB#3	Flag	ISVS FB#4	Flag	VSS TB#1	Flag	VSS TB#2	Flag
		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)		(ppbv)	
1,1,2-TRICHLOROETHANE	79-00-5	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U
TOLUENE	108-88-3	0.23	U	1.9	J	13		19		0.55	J	0.23	U
1,2-DIBROMOETHANE	106-93-4	0.28	U	0.28	U	0.28	U	0.28	U	0.28	U	0.28	U
OCTANE	111-65-9	0.52	U	0.52	U	0.52	U	0.52	U	0.52	U	0.52	U
TETRACHLOROETHYLENE	127-18-4	0.34	U	0.34	U	0.34	U	0.34	U	0.34	U	0.34	U
CHLOROBENZENE	108-90-7	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U
HEXANENITRILE	628-73-9	0.66	U	0.66	U	0.66	U	0.66	U	0.66	U	0.66	U
ETHYLBENZENE	100-41-4	0.30	U	0.30	U	0.68	J	0.75	J	0.30	U	0.30	U
P/M-XYLENE	106-42-3	0.63	U	0.63	U	2.3	J	2.5	J	0.63	U	0.63	U
CYCLOHEXANONE	108-94-1	2.9	U	2.9	U	2.9	U	2.9	U	2.9	U	2.9	U
STYRENE	100-42-5	0.33	U	0.33	U	0.33	U	0.33	U	0.33	U	0.33	U
1,1,2,2-TETRACHLOROETHANE	79-34-5	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U
O-XYLENE	95-47-6	0.40	U	0.40	U	0.40	U	0.71	J	0.40	U	0.40	U
NONANE	111-84-2	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U
1-ETHYL-2-METHYL BENZENE	611-14-3	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U
1,3,5-TRIMETHYLBENZENE	108-67-8	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U
1,2,4-TRIMETHYLBENZENE	95-63-6	0.47	U	0.47	U	0.47	U	0.47	U	0.47	U	0.47	U
DECANE	124-18-5	0.47	U	0.47	U	0.59	J	0.67	J	0.47	U	0.72	J
1,3-DICHLOROBENZENE	541-73-1	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U
1,4-DICHLOROBENZENE	106-46-7	0.43	U	0.43	U	0.43	U	0.43	U	0.43	U	0.43	U
1,2-DICHLOROBENZENE	95-50-1	0.72	U	0.72	U	0.72	U	0.72	U	0.72	U	0.72	U
UNDECANE	1120-21-4	0.63	U	0.63	U	0.63	U	0.63	U	0.63	U	0.63	U
1,2,4-TRICHLOROBENZENE	120-82-1	1.9	U	1.9	U	1.9	U	1.9	U	1.9	U	1.9	U
DODECANE	112-40-3	3.4	U	3.4	U	3.4	U	3.4	U	3.4	U	3.4	U
HEXACHLORO-1,3-BUTADIENE	87-68-3	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U
TRIDECANE	629-50-5	6.6	U	6.6	U	6.6	U	6.6	U	6.6	U	6.6	U
TETRADECANE	629-59-4	10	J	8.2	J	6.0	J	6.1	J	15	J	12	J
TBP	126-73-8	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z	<0.83	Z

Data Qualifier Flags

J Target compound detected above the IDL but below the EQL.

U Target compound not detected at or above the IDL.

Y Initial calibration and CCV was performed; however, the analyte was not part of the current operating procedure.

Z Retention time and mass spectral characteristics were determined and detectability possible at 0.8 ppbv; however, this compound is not currently part of the analytical method. See Section E.4 for more information.

Table F.8. Triple Sorbent Trap Blank Sample Analysis Results for All Target Analytes Associated with the Headspace Sampling of Tank S-102 on 4/4/96

Target Analytes	CAS No.	S6031-A12.893		S6031-A13.894		S6030-A40.761		S6030-A41.764		S6031-A14.895		S6031-A15.896	
		VSS FB#1		VSS FB#2		ISVS FB#3		ISVS FB#4		VSS TB#1		VSS TB#2	
		(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag	(ppbv)	Flag
1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE	76-14-2	0.41	U	0.41	U	0.41	U	0.41	U	0.41	U	0.41	U
VINYL CHLORIDE	75-01-4	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
METHANOL	67-56-1	<192	Y	<192	Y	<192	Y	269	Y	<192	Y	<192	Y
BUTANE	106-97-8	1.4	U	1.4	U	1.9	J	1.8	J	1.4	U	1.4	U
CHLOROETHANE	75-00-3	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U	1.4	U
ETHANOL	64-17-5	<133	Y	<133	Y	<133	Y	<133	Y	<133	Y	<133	Y
ACETONITRILE	75-05-8	1.8	U	1.8	U	7.7	J	5.0	J	1.8	U	1.8	U
ACETONE	67-64-1	16	J	15	J	28	J	43		18	J	17	J
TRICHLOROFLUOROMETHANE	75-69-4	0.72	U	0.72	U	4.3	J	115		0.72	U	1.6	J
PENTANE	109-66-0	0.89	U	0.89	U	0.89	U	0.89	U	0.89	U	0.89	U
1,1-DICHLOROETHENE	75-35-4	0.47	U	0.47	U	0.47	U	0.47	U	0.47	U	0.47	U
METHYLENE CHLORIDE	75-09-2	112		22	J	11	J	44		12	J	158	
1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE	76-13-1	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U
PROPANENITRILE	107-12-0	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U	0.77	U
PROPANOL	71-23-8	2.0	U	2.0	U	2.0	U	2.0	U	2.0	U	2.0	U
1,1-DICHLOROETHANE	75-34-3	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U
2-BUTANONE	78-93-3	2.6	J	2.6	J	3.3	J	14		2.4	J	2.3	J
CIS-1,2-DICHLOROETHENE	156-59-2	0.58	U	0.58	U	0.58	U	0.58	U	0.58	U	0.58	U
HEXANE	110-54-3	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U	0.32	U
CHLOROFORM	67-66-3	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U
TETRAHYDROFURAN	109-99-9	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
1,2-DICHLOROETHANE	107-06-2	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U
BUTANENITRILE	109-74-0	0.45	U	0.45	U	0.45	U	0.45	U	0.45	U	0.45	U
1,1,1-TRICHLOROETHANE	71-55-6	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U	0.44	U
1-BUTANOL	71-36-3	2.3	U	2.3	U	2.3	U	2.3	U	2.3	U	2.3	U
BENZENE	71-43-2	0.40	U	0.40	U	1.5	J	1.4	J	0.40	U	0.40	U
CARBON TETRACHLORIDE	56-23-5	0.20	U	0.20	U	0.20	U	0.20	U	0.20	U	0.20	U
CYCLOHEXANE	110-82-7	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U	1.1	U
1,2-DICHLOROPROPANE	78-87-5	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U
TRICHLOROETHENE	79-01-6	0.61	U	0.61	U	0.61	U	0.61	U	0.61	U	2.8	J
HEPTANE	142-82-5	0.35	U	0.35	U	2.4	J	2.6	J	0.35	U	0.35	U
4-METHYL-2-PENTANONE	108-10-1	0.46	U	1.8	J	0.46	U	0.46	U	0.46	U	0.46	U
CIS-1,3-DICHLOROPROPENE	10061-01-5	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U	0.25	U
PYRIDINE	110-86-1	6.1	U	6.1	U	6.1	U	6.1	U	6.1	U	6.1	U
TRANS-1,3-DICHLOROPROPENE	10061-02-6	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U	0.53	U
PENTANENITRILE	110-59-8	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U	0.22	U

F.16

Appendix G

Tank Vapor Characterization: Chain of Custody Sample Control Forms

Battelle Pacific
National Northwest Lab

CHAIN OF CUSTODY

WHC 100064

Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141
Page 85-3009 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437
Page 85-9656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6030
ISVS

Collection date 04 - 04 - 96
Preparation date 04 - 01 - 96

Ice Chest No.

Field Logbook No. WHC-647-A-8 ^{ADN}
N-647-8 161196

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6030 - A34 . S38
S6030 - A35 . S39
S6030 - A36 . S40

Collect NH₃/H₂O Sorbent Trap
Collect NH₃/H₂O Sorbent Trap
Collect NH₃/H₂O Sorbent Trap

S6030 - A42 . S41
S6030 - A43 . S42

Store sample bundle NH₃/H₂O field blank #1
Store sample bundle NH₃/H₂O field blank #2

[] Field Transfer of Custody			[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time	
G W Dennis <i>G.W.Dennis</i>	04 - 03 - 96	0850	J A Edwards <i>J A Edwards</i>	04 - 03 - 96	0850	
J A Edwards <i>J A Edwards</i>	04 - 3 - 96	1445	Rm Arnold <i>Rm Arnold</i>	4 - 3 - 96	1445	
Rm Arnold <i>Rm Arnold</i>	04 - 7 - 96	1530	RD Mahon <i>RD Mahon</i>	04 - 03 - 96	1530	
RD Mahon <i>RD Mahon</i>	04 - 16 - 96	0835	T.B.Utecht <i>T.B.Utecht</i>	4 - 16 - 96	0835	
T.B.Utecht <i>T.B.Utecht</i>	04 - 16 - 96	1035	JL Utecht <i>JL Utecht</i>	4 - 16 - 96	1035	
J A Edwards <i>J A Edwards</i>	4 - 17 - 96	1035	G.W. Dennis <i>G.W.Dennis</i>	4 - 17 - 96	1035	
G.W.Dennis <i>G.W.Dennis</i>	4 - 19 - 96	1330	S.O. Slate <i>Sally O. Slate</i>	4 - 19 - 96	1330	

Final Sample Disposition

Comments:

PNNL (only) Checklist	Pick-up / Delivery	Comments:
Media labeled and checked?	☒ Y / ☐ N	
Letter of instruction?	☒ Y / ☐ N	
Media in good condition?	☒ Y / ☐ N	
COC info/signatures complete?	☒ Y / ☐ N	
Rad release stickers on samples?	☐ Y / ☒ N	
Activity report from 222S?	☐ Y / ☒ N	
RSR/copy? (a ≤100/B ≤400 pCi/g)	☐ Y / ☒ N	
COC copy for LRB, RIDS filed?	☐ Y / ☒ N	

POC *(Signature)* POC *QA*

(Revised 11/30/95 PNNL)

Battelle Pacific
National Northwest Lab

CHAIN OF CUSTODY

WHC 100065

Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141

Page 85-3009 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437

Page 85-9656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6031
VSS

Collection date 04 - 04 - 96

Preparation date 04 - 01 - 96

Ice Chest No.

Field Logbook No. WHC-N-647-10

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6031 - A17 . S43

Collect NH₃/H₂O Sorbent Trap

S6031 - A18 . S44

Collect NH₃/H₂O Sorbent Trap

S6031 - A19 . S45

Collect NH₃/H₂O Sorbent Trap

S6031 - A20 . S46

Collect NH₃/H₂O Sorbent Trap

S6031 - A21 . S47

Open, close and store NH₃/H₂O field blank #1

S6031 - A22 . S48

Open, close and store NH₃/H₂O field blank #2

[] Field Transfer of Custody		[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
G W Dennis	04 - 03 - 96	0850	J A Edwards	04 - 03 - 96	0850
J A Edwards	4-3-96	1445	Rm Arnold	4-3-96	1445
Rm Arnold	4-3-96	1530	R D Mahon	04-03-96	1530
R D Mahon	04-16-96	0835	T B Lister	4-16-96	0835
T B Lister	4-16-96	1035	J A Edwards	4-16-96	1035
J A Edwards	4-17-96	1035	G W Dennis	4-16-96	1035
G W Dennis	4-18-96	1330	S O Slate	4-18-96	1330

Final Sample Disposition

Comments:

PNNL (only) Checklist	Pick-up / Delivery	Comments:
Media labeled and checked?	☒ Y / ☐ N	
Letter of instruction?	☒ Y / ☐ N	
Media in good condition?	☒ Y / ☐ N	
COC info/signatures complete?	☒ Y / ☐ N	
Rad release stickers on samples?	☐ Y / ☒ N	
Activity report from 222S?	☐ Y / ☒ N	
RSR/copy? (a ≤100/B ≤400 pCi/g)	☐ Y / ☒ N	
COC copy for LRB, RIDS filed?	☐ Y / ☒ N	

POC

UE

POC

99

(Revised 11/30/95 PNNL)

Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141
Page 85-3009 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-2891
Page 85-3656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6030
(ISVS Cart)

Collection date 04-04-96
Preparation date 04-03-96

Ice Chest No.

Field Logbook No. WHC-647-1-8 ^{km} 647-8
N-647-8

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6030 - A27 . 265 Collect Ambient Air Sample SUMMA #1

S6030 - A28 . 266 Collect SUMMA #2

S6030 - A29 . 267 Collect SUMMA #3

S6030 - A30 . 269 Collect SUMMA #4

[] Field Transfer of Custody			[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time	
J A Edwards <i>J A Edwards</i>	04-03-96	1445	RM Arnold <i>RM Arnold</i>	04-03-96	1445	
RM Arnold <i>RM Arnold</i>	04-03-96	1530	RD Mahon <i>RD Mahon</i>	04-03-96	1530	
RD Mahon <i>RD Mahon</i>	04-16-96	0835	<i>T-B Litchfield / T-B Litchfield</i>	4-16-96	0835	
<i>Tim B Litchfield / T-B Litchfield</i>	4-16-96	1035	<i>JL Juley / J L Juley</i>	4-16-96	1035	

Final Sample Disposition

Comments:

PNNL (only) Checklist	Pick-up / Delivery	Comments:
Media labeled and checked?	☒ Y / ☒ N	
Letter of instruction?	☒ Y / ☒ N	
Media in good condition?	☒ Y / ☒ N	
COC info/signatures complete?	☒ Y / ☒ N	
Rad release stickers on samples?	☒ Y / ☒ N	
Activity report from 222S?	☒ Y / ☒ N	
RSR/copy? (a ≤100/B ≤400 pCi/g)	☒ Y / ☒ N	
COC copy for LRB, RIDS filed?	☒ Y / ☒ N	

POC *[Signature]* POC *[Signature]*

(Revised 11/30/95 PNNL)

Battelle Pacific National Northwest Lab	CHAIN OF CUSTODY	WHC 100062
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Custody Form Initiator J. A. Edwards - PNNL

Telephone (509) 373-0141
Page 85-3009 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-2891
Page 85-9656 / FAX 373-3793

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6031
(VSS Truck)

Collection date 04 - 04 - 96
Preparation date 04 - 03 - 96

Ice Chest No.

Field Logbook No. WHC-N-647-10

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to PNNL

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6031 - A03 .025 ✓	Collect Ambient Air Sample SUMMA #1	Upwind of Tank
S6031 - A04 .079 ✓	Collect Ambient Air Sample SUMMA #2	
S6031 - A05 .148 ✓	Collect SUMMA #3	
S6031 - A06 .223 ✓	Collect SUMMA #4	
S6031 - A07 .231 ✓	Collect SUMMA #5	

[] Field Transfer of Custody		[X] Chain of Possession		(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time
J A Edwards <i>J A Edwards</i>	04-03-96	1445	Rm Arnold <i>Rm Arnold</i>	04-03-96	1445
Rm Arnold <i>Rm Arnold</i>	04-07-96	1530	RD Mahon <i>RD Mahon</i>	04-03-96	1530
RD Mahon <i>RD Mahon</i>	04-16-96	0835	T-B Utecht <i>T-B Utecht</i>	4-16-96	0835
T-B Utecht <i>T-B Utecht</i>	4-16-96	1035	JL Tuleya <i>JL Tuleya</i>	4-16-96	1035

Final Sample Disposition

Comments:

PNNL (only) Checklist	Pick-up / Delivery	Comments:
Media labeled and checked?	☒ Y / ☐ N	
Letter of instruction?	☒ Y / ☐ N	
Media in good condition?	☒ Y / ☐ N	
COC info/signatures complete?	☒ Y / ☐ N	
Rad release stickers on samples?	☒ Y / ☐ N	
Activity report from 222S?	☒ Y / ☐ N	
RSR/copy? (a ≤100/B ≤400 pCi/g)	☒ Y / ☐ N	
COC copy for LRB, RIDS filed?	☒ Y / ☐ N	

POC *[Signature]* POC *[Signature]*

(Revised 11/30/95 PNNL)

**Battelle Pacific
Northwest Laboratory**

CHAIN OF CUSTODY

WHC 100057

Custody Form Initiator J. A. Edwards - PNL

Telephone (509) 373-0141
Page 85-3009 / PB-08 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437
Page 85-9656 / S3-27 / FAX 373-7076

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6030
(ISVS Cart)

Collection date 04 - 07 - 96
Preparation date 04 - 01 - 96

Ice Chest No.

Field Logbook No. WHC ~~647-10-8~~ ^{R2} 16 APR 96
N-647-8

Etco Hi/Lo thermometer No. PNL-T-004

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to WHC-

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6030 - A31 . 740 PNL Triple Sorbent Trap (TST) Sample # 1
S6030 - A32 . 742 PNL TST Sample # 2
S6030 - A33 . 760 PNL TST Sample # 3

S6030 - A40 . 761 Open, close & store PNL TST Field Blank # 3
S6030 - A41 . 764 Open, close & store PNL TST Field Blank # 4

[] Field Transfer of Custody			[X] Chain of Possession			(Sign and Print Names)	
Relinquished By	Date	Time	Received By	Date	Time		
JL Julia	04-02-96	1325	JA Edwards	04-02-96	1325		
JA Edwards	04-03-96	1445	Rm Arnold	04-03-96	1445		
Rm Arnold	04-03-96	1530	RD Mahon	04-03-96	1530		
RD Mahon	04-16-96	0835	T-B. Litchfield	4-16-96	0835		
T-B. Litchfield	4-16-96	1035	JL Julia	4-16-96	1035		

Final Sample Disposition

Comments:

PNL (only) Checklist

Media labeled and checked?	Y/N
Letter of instruction?	Y/N
Media in good condition?	Y/N
COC info/signatures complete?	Y/N
Sorbents shipped on ice? (<5°C)	Y/N
Hi/Lo thermometer - <u>Keep upright!</u>	Y/N
Hi/Lo thermometer	Y/N
Rad release stickers on samples?	Y/N
Activity report from 222S?	Y/N
COC copy for LRB, RIDS filed?	Y/N

POC

POC

Comments:

Cooler Temperature Status

Hi -13 °C / Lo -13 °C (pick-up at PNL to WHC)	
Hi 17 °C / Lo -11 °C (delivery at WHC from PNL)	
Hi °C / Lo °C (at return to PNL from WHC)	
Hi °C / Lo °C (at delivery from WHC to PNL)	

(Revised 06/21/95 PNL)

**Battelle Pacific
Northwest Laboratory**

CHAIN OF CUSTODY

WHC 100058

Custody Form Initiator J. A. Edwards - PNL

Telephone (509) 373-0141
Page 85-3009 / P8-08 / FAX 376-0418

Company Contact R. D. Mahon - WHC

Telephone (509) 373-7437
Page 85-9656 / S3-27 / FAX 373-7076

Project Designation/Sampling Locations 200 West Tank Farm
241-S-102 Tank Vapor Sample SAF S6031
(VSS Truck)

Collection date 04 - 07 - 96
Preparation date 04 - 01 - 96

Ice Chest No.

Field Logbook No. WHC- N-892 10

Erteco Hi/Lo thermometer No. PNL-T-00 4

Bill of Lading/Airbill No. N/A

Offsite Property No. N/A

Method of Shipment Government Truck

Shipped to WHC-

Possible Sample Hazards/Remarks Unknown at time of sampling

Sample Identification

S6031 - A08 . 888	PNL Triple Sorbent Trap (TST) Sample # 1	
S6031 - A09 . 889	PNL TST Sample # 2	
S6031 - A10 . 890	PNL TST Sample # 3	
S6031 - A11 . 892	PNL TST Sample # 4	
S6031 - A12 . 893	Open, close & store PNL TST Field Blank # 1	In VSS truck
S6031 - A13 . 894	Open, close & store PNL TST Field Blank # 2	In VSS truck
S6031 - A14 . 895	Store PNL TST Trip Blank # 1	None
S6031 - A15 . 896	Store PNL TST Trip Blank # 2	None

[] Field Transfer of Custody			[X] Chain of Possession		(Sign and Print Names)		
Relinquished By	Date	Time	Received By	Date	Time		
JL Julia	04-02-96	1325	JA Edwards	04-02-96	1325		
JA Edwards	04-03-96	1445	R.D. Mahon	04-03-96	1445		
R.D. Mahon	04-03-96	1530	R.D. Mahon	04-03-96	1530		
R.D. Mahon	04-16-96	0835	Tim Blumenthal	4-16-96	0835		
Tim Blumenthal	4-16-96	1035	JL Julia	4-16-96	1035		

Final Sample Disposition

Comments:

PNL (only) Checklist

Media labeled and checked?	Y/N	Pick-up / Delivery
Letter of instruction?	Y/N	
Media in good condition?	Y/N	Y/N
COC info/signatures complete?	Y/N	Y/N
Sorbents shipped on ice? (<5°C)	Y/N	Y/N
Hi/Lo thermometer - <u>Keep upright!</u>	Y/N	
Hi/Lo thermometer		Y/N
Rad release stickers on samples?		Y/N
Activity report from 222S?		Y/N
COC copy for LRB, RIDS filed?		Y/N

POC (12) POC 49

Comments:

Cooler Temperature Status

Hi	-13 °C / Lo	-13 °C (pick up at PNL to WHC)
Hi	-17 °C / Lo	-11 °C (delivery at WHC from PNL)
Hi	°C / Lo	°C (at return to PNL from WHC)
Hi	°C / Lo	°C (at delivery from WHC to PNL)

(Revised 06/21/95 PNL)

Distribution List

PNNL-11270

PNNL

Karl Pool	P8-08
Berta Thomas	P8-08
John Evans	K6-96
Khris Olsen	K6-96
Kurt Silvers	K9-08
Jon Fruchter	K6-96
Jim Huckaby	K6-80
Brenda Thornton	K6-80
Darlene Varley	K1-06
Katherine Savard	K9-04
Kris Walters	K6-80 (5 copies)

Lockheed

Larry Pennington	S7-21
Luther Buckley	R2-12

DOE-RL

Carol Babel	S7-54
Jim Thompson	S7-54